An Investigation to Locate and Correct Errors in the Card File Prepared by the Joint Committee on Powder Diffraction Standards (JCPDS)

by

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INTRODUCTION

The main consideration of this research was to search for, find, and where possible correct errors in the card file of X-ray diffraction data prepared by the Joint Committee on Powder Diffraction Standards (JCPDS). Errors, if any, are located using a non-linear least-squares program prepared by C.E. Corbato. Potiental errors expected to occur include incorrect indexing of reflections, incorrect d-values and/or cell dimensions, incorrect reflections attributable to impurities, typographical errors, or a combination of these.

NON-LINEAR LEAST-SQUARES METHOD

The method of least squares is applied to cell parameter refinement in this research, but it doesn't always give the best answer. The method is based on the premise that the variable for which the sum of the squares of the residuals to be minimized is distributed about its mean value. In X-ray diffraction work this value is theta or 2-theta. Unfortunately, the cell parameters are not linearly related to theta which requirs a non-linear least-squares procedure, A very accurate first approximation of the refined cell parameters may be obtained by first refining on the sum of the squares of $1/d^2$.

LEAST-SQUARES REFINEMENT OF CELL PARAMETERS

TETRAGONAL AND HEXAGONAL

THEORY

Consider the d-spacing formulas:

(eqn. 1) $$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \text{ (tetragonal)},$$

$$\frac{1}{d^2} = \frac{4(h^2+hk+k^2)}{3a^2} + \frac{l^2}{c^2} \text{ (hexagonal)}.$$

(h,k, and l are Miller indices of the diffracting planes, d is the interplaner spacing and a and c are unit cell dimensions)

Let $D=\frac{1}{d^2}$, $A=\frac{1}{a^2}$, $C=\frac{1}{c^2}$, $r=h^2+k^2$ or $\frac{4}{3}(h^2+hk+k^2)$, $s=l^2$.

Then eqn. 1 becomes a linear equation in two variables, A and C:

(eqn. 2) $$D=rA+sC$$

The "normal" equation for least-squares solution of this linear equation are the following:

$$\left[\Sigma r_i^2\right]A + \left[\Sigma r_i s_i\right]C = \Sigma r_i D_i$$

$$\left[\Sigma r_i s_i\right]A + \left[\Sigma s_i^2\right]C = \Sigma s_i D_i$$

Solution of these two equations yields A and C, from which a and c can be obtained. Let the matrix

$B_{ij}=\begin{bmatrix} \Sigma r^2 & \Sigma rs \\ \Sigma rs & \Sigma s^2 \end{bmatrix}$ and B_{ij}^{-1} its inverse so that $B \cdot B^{-1} = 1$.

The variance of the unknowns, A and C, is then given by the following:

$$\sigma_A^2 = B_{11}^{-1}\left(\frac{f}{n-2}\right), \quad \sigma_C^2 = B_{22}^{-1}\left(\frac{f}{n-2}\right)$$

where f is the sum of the squares of the "residuals", i.e., the differences between D_o (derived from the observed d's) and D_c (calculated using the values of A and C). From these relationships, it is possible to determine the errors of the cell parameters, σ_a and σ_c, using the equations

$$\sigma_a^2 = \left(\frac{2a}{2A}\right)^2 \sigma_A^2 \text{ and } \sigma_c^2 = \left(\frac{2c}{2C}\right)\sigma_C^2,$$

so that

$$\sigma_a = \pm\tfrac{1}{2}\left[\frac{B_{11}^{-1} f}{A^3(n-2)}\right]^{\frac{1}{2}} \text{ and } \sigma_c = \pm\tfrac{1}{2}\left[\frac{B_{22}^{-1} f}{C^3(n-2)}\right]^{\frac{1}{2}}.$$

METHOD

1. Enter n values of hkl and d_o (observed).
2. Calculate n values of r, s, and D_o.
3. Calculate the five sums which are functions of r, s, and D_o and enter into matrix (1).
4. Solve for unknowns and inverse of matrix (using Gauss-Jordan method):
    a. Divide each term of the first row of matrix (1) by B_{11} and place quotients in 1st row of matrix (2); divide each term of 2nd row of matrix (1) by B_{21} and place quotients in 2nd row of matrix (2).
    b. Rewrite 1st row of matrix (2) in the 1st row of matrix (3); subtract terms of first

row of matrix (2) from corresponding terms of 2nd row and place in 2nd row of matrix (3).

c. Divide each term in 1st row of matrix (3) by the term now in B_{12} position of matrix (3) and place quotients in 1st row of matrix (4); divide each term in 2nd row of matrix (3) by the term now in B_{22} position of matrix (3) and place quotients in 2nd row of matrix (4).

d. Subtract terms of 2nd row of matrix (4) from corresponding terms of 1st row and place in 1st row of matrix (5); rewrite 2nd row of matrix (4) in 2nd row of matrix (5) and of matrix (6).

5. Calculate n values of D_c(calculated), Δ, and d_c.

6. Calculate f.

7. Calculate a, c, σ_a, and σ_c.

—— All work should be recorded to at least 5 significant figures; results of a, c, σ_a, and σ_c should be given to 3 figures to the right of the decimal point.

Worksheet for Least-Squares Refinement of Cell Parameters

Substance: Portlandite - $Ca(OH)_2$ System: Hexagonal

hkl	d_o	r	s	D_o	D_c	Δ	d_c
001	4.90	0	1	.041649	.041623	.000026	4.902
100	3.112	4/3	0	.10326	.10318	.00008	3.113
101	2.628	4/3	1	.14479	.14480	−.00001	2.628
002	2.447	0	4	.16701	.16649	.00052	2.451
102	1.927	4/3	4	.26930	.26967	−.00037	1.926
110	1.796	4	0	.31002	.30954	.00048	1.797
111	1.687	4	1	.35137	.35116	.00021	1.688
003	1.634	0	9	.37454	.37461	−.00007	1.634
200	1.557	16/3	0	.41250	.41272	−.00022	1.557
201	1.484	16/3	1	.45408	.45434	−.00026	1.484

$r = h^2 + k^2$ (tetragonal)
$= 4/3(h^2 + hk + k^2)$ (hexagonal)
$s = l^2$
$D_o = 1/d_o^2$

$D_c = rA + sC$
$\Delta = D_o - D_c$
$d_c = (1/D_c)^{1/2}$

$\Sigma r^2 =$ 94.222 $\Sigma rD_o =$ 7.9571
$\Sigma rs =$ 16.000 $\Sigma sD_o =$ 6.1080
$\Sigma s^2 =$ 117.00 $f = \Sigma\Delta^2 =$ 8.10×10^{-7}

B_{ij}

(1)

$\Sigma r^2 =$ 94.222	$\Sigma rs =$ 16	$\Sigma rD_o =$ 7.9571	1	0
$\Sigma rs =$ 16	$\Sigma s^2 =$ 117	$\Sigma sD_o =$ 6.1080	0	1

(2)

1	.16981	.084451	.010613	0
1	7.3125	.38175	0	.062500

(3)

1	.16981	.084451	.010613	0
0	7.1427	.29730	−.010613	.062500

(4)

5.8889	1	.49733	.062499	0
0	1	.041623	−.0014859	.0087502

(5)

5.8889	0	.45571	.063985	−.0087502
0	1	.041623	−.0014859	.0087502

(6)

1	0	$A =$ .077385	$B_{11}^{-1} =$ .010865	$B_{12}^{-1} =$ −.0014859
0	1	$C =$ .041623	$B_{21}^{-1} =$ −.0014859	$B_{22}^{-1} =$ .0087502

B_{ij}^{-1}

Checks: $B_{12}^{-1} = B_{21}^{-1}$ ✓

$B_{11} \cdot B_{11}^{-1} + B_{12} \cdot B_{21}^{-1} =$ ✗ .99995

$B_{21} \cdot B_{12}^{-1} + B_{22} \cdot B_{22}^{-1} = 1$ ✓

$a = (1/A)^{1/2}$, $\sigma_a = \pm\frac{1}{2}\left[\frac{B_{11}^{-1} f}{A^3 (n-2)}\right]^{1/2}$

$c = (1/C)^{1/2}$, $\sigma_c = \pm\frac{1}{2}\left[\frac{B_{22}^{-1} f}{C^3 (n-2)}\right]^{1/2}$

$a =$ 3.595 ± .001

$c =$ 4.902 ± .002

Worksheet for Least-Squares Refinement of Cell Parameters

Substance: THORITE System: TETRAGONAL

hkl	d_o	r	s	D_o	D_c	Δ	d_c
101	4.72	1	1	.044887	.04481	.00008	4.72403
200	3.55	4	0	.079349	.07896	.00039	3.55874
211	2.842	5	1	.123809	.12377	.00004	2.84245
112	2.676	2	4	.139646	.13976	-.00011	2.67491
220	2.516	8	0	.157971	.15792	.00005	2.51641
202	2.361	4	4	.179394	.17924	.00015	2.36201
301	2.222	9	1	.202541	.20273	-.00019	2.22096
103	2.019	1	9	.245317	.24537	-.00005	2.01878

$r = h^2+k^2$ (tetragonal) $\quad D_c = rA + sC \quad \Sigma r^2 = 208 \quad \Sigma rD_o = 5.31015$

$= 4/3(h^2+hk+k^2)$ (hexagonal) $\quad \Delta = D_o - D_c \quad \Sigma rs = 48 \quad \Sigma sD_o = 3.85525$

$s = l^2 \quad d_c = \left(1/D_c\right)^{1/2} \quad \Sigma s^2 = 116 \quad f = \Sigma\Delta^2 = 2.358 \times 10^{-7}$

$D_o = 1/d_o^2$

B_{ij}

(1)	$\Sigma r^2 = 208$	$\Sigma rs = 48$	$\Sigma rD_o = 5.31015$	1	0
	$\Sigma rs = 48$	$\Sigma s^2 = 116$	$\Sigma sD_o = 3.85525$	0	1
(2)	1	.23077	.02553	.00481	0
	1	2.41667	.08032	0	.02083
(3)	1	.23077	.02553	.00481	0
	0	2.18590	.05479	-.00481	.02083
(4)	4.33332	1	.11063	.02084	0
	0	1	.02507	-.00220	.00953
(5)	4.33332	0	.08656	.02304	-.00953
	0	1	.02507	-.00220	.00953
(6)	1	0	$A = .01974$	$B_{11}^{-1} = .00532$	$B_{12}^{-1} = -.00220$
	0	1	$C = .02507$	$B_{21}^{-1} = -.00220$	$B_{22}^{-1} = .00953$

B_{ij}^{-1}

$$a = \left(\frac{1}{A}\right)^{1/2}, \quad \sigma_a = \pm\frac{1}{2}\left[\frac{B_{11}^{-1} f}{A^3 (n-2)}\right]^{1/2}$$

$$c = \left(\frac{1}{C}\right)^{1/2}, \quad \sigma_c = \pm\frac{1}{2}\left[\frac{B_{22}^{-1} f}{C^3 (n-2)}\right]^{1/2}$$

$a = 7.1175 \pm .00260$

$c = 6.3157 \pm .00244$

Checks: $B_{12}^{-1} = B_{21}^{-1}$

$B_{11} \cdot B_{11}^{-1} + B_{12} \cdot B_{21}^{-1} = 1.00096$

$B_{21} \cdot B_{12}^{-1} + B_{22} \cdot B_{22}^{-1} = 1 \ (.99988)$

PROCEDURE

The procedure used in this investigation was as follows: first, a mineral was chosen from the Mineralogy Museum's mineral list which is a member of either the isometric, tetragonal, hexagonal, or orthorhombic crystal systems. The reason for only choosing minerals which are in the museum is that a mineral may need to be available in case an X-ray diffraction pattern were necessary to determine and/or correct an error in the JCPDS card file. The four crystal systems were used because the present non-linear least-squares program can only handle these four systems. Next, the JCPDS card was found for the chosen mineral and all the d-values and the corresponding hkl values were transfered from the JCPDS card to the non-linear least-squares program. The residuals of 2-theta are then calculated and compared. If there is a discrepancy of greater than 0.3° then the line is considered an error. In this case an hkl generator program is run which determines the Laue Class from the space group and gives all possible hkl values and their corresponding d-values for a mineral of that space group (input into the hkl generator program includes the space group number and the cell dimensions taken from the JCPDS card). At this point the d-spacing for which the residuals of 2-theta indicate an error is matched to its closest d-value from the hkl

generator and the corresponding hkl values are compared. If these differ then it is possible that the line was incorrectly indexed on the JCPDS card, which was checked by imputting the new hkl value into the non-linear least-squares program. If the residual of 2-theta was now less than 0.3 it was assumed that the line was incorrectly indexed and that the new hkl value was the better choice for that line. However, in a few cases the residual of 2-theta indicated no difference, indicated a larger discrepancy, or the hkl generator indicated that the original hkl was the better choice for that particular d-spacing. When one of these problems occured an X-ray diffraction pattern was run. Values of 2-theta are read directly from the X-ray diffraction pattern and are input into the non-linear least-squares program. If the output indicated that the residual of 2-theta, for the line in error, was now considerably less than the original value then it was assumed that the JCPDS value of 2-theta was incorrectly read on the original diffraction pattern. Unfortunately, if the line still gave a residual of 2-theta the same or greater than the original then no reasonable correction could be offered.

MINERAL ANALYSIS

This paper analysed the following minerals: pabstite, pyrolusite, rutile, sulfur, sylvite, tetradymite, topaz, uraninite, vanadinite, wavellite, witherite, wulfenite, zircon. Each of these thirteen minerals was investigated according to the preceeding sections.

PABSTITE

Pabstite's chemical formula is $BaSnSi_3O_9$, a barium tin silicate belonging to the Benitoite Group, and is a member of the hexagonal crystal system. The diffraction work was done on a natural pabstite mineral, collected from Santa Cruz, California, in 1965 using CuK alpha radiation with a wavelength of 1.542A and with a nickel filter. The non-linear least-squares output for pabstite is given in Table 1, page 23 . The residuals of 2-theta indicate that all lines fit well except for the 211 and the 213 lines which show deviations of .433° and .711° between the calculated and observed 2-theta values. the output also indicated that the calculated cell dimensions of a=6.7401A $\pm$ 0.0069 and c= 9.8656A $\pm$ 0.014 do not agree with the cell dimensions a= 6.724A and c= 9.854A listed on the JCPDS card (18-196). An hkl generator was run (Table 2, page 24). The hkl generator output indicated that the 211 and the 213 lines were correctly indexed. An X-ray pattern would have been run to try and correct the problem, but

the Mineral Museum doesn't contain pabstite; therefore, no further work could be done.

PYROLUSITE

The chemical formula for pyrolusite is MnO_2, a manganese oxide, and it is a member of the tetragonal crystal system. The diffraction work was done on a sythetic mineral obtained from the Baker Chemical Co., Phillipsburg, N.J. It was X-rayed at a temperture of 25°C using CuK alpha 1 radiation with a wavelength of 1.54056A and with a nickel filter. The non-linear least-squares output for pyrolusite is given in Table 3, page 25. The residuals of 2-theta indicate that all lines fit well with the largest discrepancy being 0.029°. The calculated cell dimensions a= 4.3998A $\pm$ 0.0001 and c= 2.8741A $\pm$ 0.0001 agree well with the cell dimensions, a= 4.3999A and c= 2.8740A, listed on the JCPDS card (24-735). The JCPDS card for pyrolusite contains no errors.

RUTILE

Rutile's chemical formula is TiO_2, a titanium oxide belonging to the Rutile Group, and is a member of the tetragonal crystal system. The diffraction work was done in 1969 at a temperture of 25°C using CuK alpha 1 radiation with a wavelength of 1.54056A and with a nickel filter. The non-linear least-squares output for rutile is given in Table 4, page 26. The residuals of 2-theta indicate that all

lines fit very well with the greatest discrepancy being 0.032°. The calculated cell dimensions also agree very well with the cell dimensions given on the JCPDS card (21-1276). Obviously, the JCPDS card for rutile contains no errors.

Calculated cell dimensions (Rutile)

a= 4.5934A ± 0.0001
c= 2.9592A ± 0.0001

JCPDS card cell dimensions (Rutile)

a= 4.5933A
c= 2.9592A

SULFUR

The chemical formula for sulfur is S and it is a member of the orthorhombic crystal system. The diffraction work was validated by calculated pattern 24-733. The non-linear least-squares output is given in Table 5, page 27. The residuals of 2-theta indicate that all lines fit well with the exception of the 355 line which has a discrepancy of 0.125°. An hkl generator was run and the output is given in Table 6, page 28. The output indicates that an hkl value of 515 is a better choice. The 355 line is then replaced with the new value of 515 in the non-linear least-squares program and the output is given in Table 7, page 29. The residuals of 2-theta agree well now with the largest discrepancy being 0.075°. The calculated cell dimensions and the cell dimensions given on the JCPDS cards (8-247 & 8-247A)

now fit very well. This was taken as evidence that the JCPDS card for sulfur was in error and that the hkl value of 355 should be replaced by the hkl value of 515.

JCPDS card cell dimensions (sulfur)

a= 10.45A
b= 12.84A
c= 24.46A

Calculated cell dimensions (sulfur)

a= 10.4553A $\pm$ 0.0025
b= 12.8480A $\pm$ 0.0043
c= 24.4683A $\pm$ 0.0063

SYLVITE

The chemical formula for sylvite is KCl, a potassium chloride belonging to the halite-galena-periclase group, and it is a member of the isometric crystal system. The diffraction work was done in 1953 at a temperture of 25°C using CuK alpha 1 radiation with a wavelength of 1.5405A and with a nickel filter. The non-linear least-squares output is given in Table 8, page 30. The residuals of 2-theta indicate that all lines fit very well with the greatest discrepancy being 0.038°. The output also exhibits the close correlation of the cell dimensions calculated here, a= 6.2931A $\pm$ 0.0002, and the JCPDS card (4-587) cell dimension, a= 6.2931A. It is clearly apparent that the JCPDS card for sylvite contains no errors.

TETRADYMITE

The chemical formula for tetradymite is $Bi_2Te_{1.65}S_{1.35}$, a bismuth tellurium sulfide which belongs to the Tetradymite Group, and it is a member of the hexagonal crystal system. The diffraction work was completed in 1967 on a sample collected from Paonia, Colorado using CuK alpha radiation with a wavelength of 1.5418A and with a nickel filter. The non-linear least-squares output for tetradymite is given in Table 9,page 31. The residuals of 2-theta indicate notable discrepancies in over 50% of the lines. Also, the calculated cell dimensions and the JCPDS card (19-1330) cell dimensions do not agree. Therefore, an hkl generator was run for tetradymite. The output is given in Table 10, page 32. The hkl generator indicates that all lines which show a notable discrepancy are correctly indexed. Because the discrepancies were less than 0.3°, an X-ray diffraction pattern was not run.

Calculated cell dimensions (Tetradymite)

a= 4.2349A $\pm$ 0.0010
c= 29.5603A $\pm$ 0.0080

JCPDS card cell dimensions (Tetradymite)

a= 4.2381A
c= 29.5890A

TOPAZ

The chemical formula for topaz is $Al_2SiO_4(F,OH)_2$, an aluminum silicate, and it is a member of the orthorhombic crystal system. Three different sources were used for the diffraction work.

SOURCE	GREATEST IMPURITY
Minas Gerais, Brazil	.01-.1% Fe
Durango, Mexico	.01-.1% Fe
Thomas Mountains, Utah	.01-.1% Fe

The diffraction work was done in 1962 at a temperture of 26°C using CuK alpha 1 radiation with a wavelength of 1.5405A with a nickel filter. The non-linear least-squares output is given in Table 11, page 33. The residuals of 2-theta indicate that all lines correlate very well with the largest discrepancy being 0.037°. The calculated and the JCPDS cell dimensions also agree very well; therefore it was concluded that, the JCPDS card (12-765 & 12-765A) for topaz contained no errors.

CALCULATED CELL DIMENSIONS (TOPAZ)

a= 8.3942A $\pm$ 0.0008
b= 8.7925A $\pm$ 0.0006
c= 4.6486A $\pm$ 0.0003

JCPDS CARD CELL DIMENSIONS (TOPAZ)

a= 8.394A
b= 8.792A
c= 4.649A

URANINITE

The chemical formula for uraninite is UO_2, an uranium oxide, and it is a member of the isometric crystal system. The diffraction work was done in 1953 at a temperture of 26°C using CuK alpha radiation with a wavelength of 1.5405A with a nickel filter. The non-linear least-squares output is given in table 12, page 34. The residuals of 2-theta indicate that all lines fit very well with the largest discrepancy being 0.053°. The calculated cell dimension, a= 5.4683A $\pm$ 0.0002, agrees very well with the JCPDS card (5-550) cell dimension, a= 5.4682A. Obviously, the JCPDS card for uraninite contains no errors.

VANADINITE

The chemical formula for vanadinite is $Pb_5(VO_4)_3Cl$, a lead vanadium oxide chloride which belongs to the Apatite Group, and it is a member of the hexagonal crystal system. The diffraction work was done on a sample collected from the Apache Mine near Globe, Arizona. The sample was diluted with five to seven parts by volume with gum tragacanth, then X-rayed using CuK alpha radiation with a wavelength of 1.5418A with a nickel filter. The non-linear least-squares output is given in Table 13, page 35. The residuals of 2-theta indicate that the JCPDS card (13-585) for vanadinite may contain errors. Therefore, an hkl generator was run. The hkl generator output is given in Table 14, page 36.

After close comparison, eight d-values were found to be possible errors. These eight values were re-indexed using the hkl generator output and the new values were input into the non-linear least-squares program. The output is given in Table 15, page 37. The residuals of 2-theta now indicate an exceptionally good fit of all re-indexed lines. Close agreement between calculated and JCPDS cell dimensions after the lines in error were re-indexed indicates that the JCPDS data contained misindexed reflections.

Replaced hkl	Corresponding d-value	New hkl
102	3.38	210
103	2.351	131
104	1.789	500
313	1.737	501
204	1.692	420
403	1.647	421
214	1.610	502
323	1.566	304

Calculated cell dimensions (Vanadinite)

a= 10.3317A $\pm$ 0.0033
c= 7.3446A $\pm$ 0.0041

JCPDS card cell dimensions (Vanadinite)

a= 10.331A
c= 7.343A

WAVELLITE

The chemical formula for wavellite is $Al_3(PO_4)_2(OH)_3 \cdot 5H_2O$, an aluminum phosphate hydroxide hydrate, and it is a member of the orthorhombic crystal system. Diffraction work was done on a sample collected from the

Earr Sandstone, near Black River Falls, Jackson Co., Wisconsin. FeK alpha radiation, with a wavelength of 1.9373A with a manganese filter, was used in 1972 for the diffraction work. The non-linear least-squares is given in Table 16, page 38. The residuals of 2-theta indicate that all lines fit exceptionally well with the largest discrepancy being 0.052°. The calculated and the JCPDS card (25-20 & 25-20A) cell dimensions also agree. The JCPDS card for wavellite contains no errors.

Calculated cell dimensions (Wavellite)

a= 9.6236A ± 0.002
b= 17.3370A ± 0.0036
c= 6.9849A ± 0.0014

JCPDS card cell dimensions (Wavellite)

a= 9.624A
b= 17.338A
c= 6.986A

WITHERITE

The chemical formula for witherite is $BaCO_3$, a barium carbonate belonging to the Aragonite Group, and it is a member of the orthorhombic crystal system. The diffraction work was done in 1953 at a temperture of 25°C using CuK alpha 1 radiation with a wavelength of 1.5405A and with a nickel filter. The non-linear least-squares output is given in Table 17, page 39. The residuals of 2-theta indicate that the JCPDS card (5-378) for witherite contains a number of errors. Therefore, an hkl generator was run. The output is given in Table 18, page 40. Eleven lines appear

to be misindexed as indicated by the hkl generator. New hkl values were input into the non-linear least-squares program (outout given in Table 19, page 41). The residuals of 2-theta indicate that all lines reindexed are better choices; however, one residual of 2-theta, the 022 line, displays a discrepancy of 0.248°. For this reason, an X-ray diffraction pattern was run (Fig. 1, page 45). Thirty-eight values of 2-theta were input into the non-linear least-squares program. The output is given in Table 20, page 42. The residuals of 2-theta indicate that the 022 line is still an error. The changes indicated for witherite are as follows:

Replaced hkl	Corresponding d-value	New hkl
012	3.025A	121
130	2.590A	022
310	1.737A	033
133	1.649A	241
043	1.543A	104
330	1.521A	114
313	1.348A	062
242	1.335A	243
243	1.248A	234
025	1.233A	351
312	1.215A	171

Calculated cell dimensions (Witherite)

a= 5.3125A ± 0.0024
b= 8.9034A ± 0.0031
c= 6.4367A ± 0.0028

JCPDS card cell dimensions (Witherite)

a= 5.314A
b= 8.904A
c= 6.430A

WULFENITE

Wulfenite's chemical formula is $PbMoO_4$, a lead molybdenum oxide, and it is a member of the tetragonal crystal system. The diffraction work for wulfenite (done in 1957) was completed on a sample precipitated from solutions of $PbCl_2$ and Na_2MoO_4 and then annealed at 400°C for two hours. The radiation used was CuK alpha with a wavelength of 1.5405A with a nickel filter. The non-linear least-squares output for Wulfenite is given in Table 21, page 43. The residuals of 2-theta indicate that all lines fit very well with the largest discrepancy being 0.058°. The calculated and JCPDS card (8-475) cell dimensions also agree. Therefore, the JCPDS card for wulfenite contains no errors.

Calculated cell dimensions (wulfenite)

a= 5.4347A ± 0.0003
c= 12.1104A ± 0.0013

JCPDS card cell dimensions (Wulfenite)

a= 5.435A
c= 12.11A

ZIRCON

The chemical formula for zircon is $ZrSiO_4$, a zirconium silicate, and it is a member of the tetragonal crystal system. Diffraction work for zircon was completed in 1955 using CuK alpha 1 radiation with a wavelength of 1.5405A and with a nickel filter. The non-linear least-squares output for Zircon is given in Table 22, page 44.

The small discrepancy (0.050° max.) of the residuals of 2-theta and the close correlation between the calculated and JCPDS card (6-266) cell dimensions indicates that there are no errors on the JCPDS card for zircon.

Calculated cell dimensions (Zircon)

a= 6.6037A ± 0.0003
c= 5.9791A ± 0.0004

JCPDS card cell dimensions (Zircon)

a= 6.604A
c= 5.979A

CONCLUSIONS

In this investigation were found to fit into three categories. These categories are:

1). Those minerals whose JCPDS card contained no errors.

2). Those minerals whose JCPDS card contained only minor errors.

3). Those minerals whose JCPDS card contained major errors.

Pyrolusite, rutile, sylvite, topaz, uraninite, wavellite, wulfenite, and zircon fit into the first category. Sulfur fit into the second category with only one line in error, which was corrected by reindexing using the hkl generator. The third category containes four minerals, pabstite, tetradymite, vanadinite, and witherite. Pabstite has two lines in error which could not be corrected by the hkl generator, and with no sample in the Mineralogy Museum, no further work could be done. On the other hand, tetradymite displays notable discrepancies in over 50% of its lines. However, the hkl generator indicates that the lines are correctly indexed for the 2-theta's given on the JCPDS card. Vanadinite showed errors in eight of its lines, which were corrected by reindexing them with the help of the hkl gereator program. Witherite appeared to have eleven lines in error and after reindexing, with the help of the hkl generator, only one line still indicated a notable discrepancy. This line is the 022

line which shows a discrepancy of 0.248. Because of this discrepancy, an X-ray diffraction pattern was run. After imputting the 2-theta values from the diffraction pattern into the non-linear least-squares program, the residuals of 2-theta indicated that the line was still in error.

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 6.7401A C= 9.8656A
+/- 0.0069 0.0140

SSR (DEGREES 2-THETA): 7.5111D-01
RMS RESIDUALS (DEGREES 2-THETA): 1.6997D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	0	15.132	5.8500	5.8371	-0.00013	-0.034	1.00
0	0	2	17.978	4.9300	4.9328	0.00005	0.010	1.00
1	0	2	23.579	3.7700	3.7676	-0.00009	-0.015	1.00
1	1	0	26.426	3.3700	3.3700	0.00000	0.000	1.00
1	1	1	27.946	3.1900	3.1891	-0.00005	-0.008	1.00
2	0	0	30.623	2.9170	2.9185	0.00012	0.017	1.00
1	1	2	32.124	2.7840	2.7826	-0.00013	-0.016	1.00
2	0	2	35.714	2.5120	2.5118	-0.00002	-0.003	1.00
0	0	4	36.418	2.4650	2.4664	0.00019	0.021	1.00
1	0	4	39.618	2.2730	2.2719	-0.00019	-0.020	1.00
2	1	0	40.874	2.2060	2.2062	0.00004	0.004	1.00
2	1	1	42.359	2.1320	2.1530	0.00428	0.433	1.00
2	1	2	44.973	2.0140	2.0140	-0.00001	-0.001	1.00
1	1	4	45.570	1.9890	1.9903	0.00033	0.032	1.00
3	0	0	46.534	1.9500	1.9457	-0.00116	-0.109	1.00
2	0	4	48.293	1.8830	1.8838	0.00024	0.022	1.00
2	1	3	49.013	1.8570	1.8321	-0.00793	-0.711	1.00
3	0	2	50.344	1.8110	1.8100	-0.00034	-0.030	1.00
2	2	0	54.510	1.6820	1.6850	0.00127	0.106	1.00
0	0	6	55.916	1.6430	1.6443	0.00057	0.047	1.00
3	1	0	56.820	1.6190	1.6189	-0.00004	-0.003	1.00
3	1	1	57.794	1.5940	1.5976	0.00175	0.141	1.00
1	0	6	58.315	1.5810	1.5827	0.00084	0.067	1.00
3	1	2	60.067	1.5390	1.5382	-0.00044	-0.035	1.00
3	0	4	60.588	1.5270	1.5276	0.00033	0.026	1.00
1	1	6	62.868	1.4770	1.4778	0.00047	0.036	1.00

Table 1. Pabstite non-linear least-squares output

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	5.8232												
0	0	2	4.9270												
1	0	2	3.7613												
1	1	0	3.3620												
1	1	1	3.1819												
2	0	0	2.9116												
1	1	2	2.7771												
2	0	2	2.5066												
0	0	4	2.4635												
1	1	3	2.3495												
1	0	4	2.2688												
2	1	0	2.2009												
2	1	1	2.1480												
2	1	2	2.0096												
1	1	4	1.9871												
3	0	0	1.9411												
2	0	4	1.8806												
2	1	3	1.8284												
3	0	2	1.8060												
1	1	5	1.7002												
2	2	0	1.6810												
2	2	1	1.6571												
0	0	6	1.6423												
2	1	4	1.6413												
3	1	0	1.6151												
3	1	1	1.5938												
2	2	2	1.5910												
1	0	6	1.5807												
3	1	2	1.5347												
3	0	4	1.5246												
2	2	3	1.4964												
1	1	6	1.4757												
2	1	5	1.4682												
4	0	0	1.4558												
3	1	3	1.4493												
2	0	6	1.4305												

Table 2. hkl generator (Pabstite)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 4.3998A C= 2.8741A
+/- 0.0001 0.0001

SSR (DEGREES 2-THETA): 4.2284D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.2514D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	0	28.680	3.1100	3.1111	0.00008	0.011	1.00
1	0	1	37.328	2.4070	2.4062	-0.00012	-0.013	1.00
2	0	0	41.010	2.1990	2.1999	0.00017	0.018	1.00
1	1	1	42.823	2.1100	2.1111	0.00024	0.024	1.00
2	1	0	46.081	1.9681	1.9677	-0.00012	-0.011	1.00
2	1	1	56.452	1.6234	1.6236	0.00010	0.008	1.00
2	2	0	59.370	1.5554	1.5556	0.00009	0.007	1.00
0	0	2	64.828	1.4370	1.4370	0.00002	0.001	1.00
3	1	0	67.240	1.3912	1.3913	0.00011	0.008	1.00
2	2	1	68.554	1.3677	1.3680	0.00027	0.020	1.00
3	0	1	72.260	1.3064	1.3064	-0.00004	-0.003	1.00
1	1	2	72.382	1.3045	1.3046	0.00007	0.005	1.00
3	1	1	75.910	1.2524	1.2523	-0.00008	-0.006	1.00
2	0	2	79.636	1.2029	1.2031	0.00022	0.015	1.00
3	1	2	83.182	1.1604	1.1605	0.00011	0.008	1.00
3	2	1	86.596	1.1232	1.1232	0.00005	0.004	1.00
4	0	0	88.895	1.1000	1.1000	-0.00007	-0.005	1.00
2	2	2	93.724	1.0556	1.0556	-0.00008	-0.005	1.00
3	3	0	95.940	1.0370	1.0370	0.00009	0.006	1.00
3	1	2	100.786	0.9998	0.9996	-0.00042	-0.029	1.00
4	2	0	103.066	0.9838	0.9838	0.00006	0.004	1.00
1	0	3	110.762	0.9360	0.9361	0.00020	0.015	1.00
4	0	2	123.753	0.8734	0.8734	0.00015	0.012	1.00
3	1	0	126.420	0.8629	0.8629	-0.00007	-0.006	1.00
3	1	3	126.817	0.8614	0.8613	-0.00016	-0.014	1.00
4	1	2	129.087	0.8567	0.8567	0.00010	0.008	1.00
3	3	2	132.671	0.8410	0.8409	-0.00021	-0.020	1.00

Table 3. Pyrolusite non-linear least-squares output

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 4.5953A C= 2.9067A
+/- 0.0374 0.0524

SSR (DEGREES 2-THETA): 1.5723D+03
RMS RESIDUALS (DEGREES 2-THETA): 6.6088D+00

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	0	27.446	3.2470	3.2494	0.00014	0.020	1.00
1	0	1	36.085	2.4870	2.4565	-0.00404	-0.463	1.00
2	0	0	39.187	2.2970	2.2976	0.00011	0.012	1.00
1	1	1	41.225	2.1880	2.1664	-0.00419	-0.430	1.00
2	1	0	44.050	2.0540	2.0551	0.00025	0.024	1.00
2	1	1	54.322	1.6874	1.6780	-0.00393	-0.328	1.00
2	2	0	56.640	1.6237	1.6247	0.00046	0.037	1.00
0	0	2	62.740	1.4797	1.4534	-0.01671	-1.271	1.00
3	1	0	64.039	1.4528	1.4532	0.00024	0.018	1.00
2	2	1	65.478	1.4243	1.4182	-0.00426	-0.318	1.00
3	0	1	69.008	1.3598	1.3551	-0.00374	-0.272	1.00
1	1	2	69.788	1.3465	1.3267	-0.01659	-1.198	1.00
3	1	1	72.408	1.3041	1.2998	-0.00391	-0.279	1.00
2	0	2	76.508	1.2441	1.2283	-0.01677	-1.170	1.00
2	1	2	79.819	1.2006	1.1866	-0.01646	-1.135	1.00
3	2	1	82.333	1.1702	1.1672	-0.00372	-0.255	1.00
4	0	0	84.258	1.1483	1.1488	0.00069	0.047	1.00
4	1	0	87.461	1.1143	1.1145	0.00032	0.022	1.00
2	2	2	89.555	1.0936	1.0832	-0.01613	-1.097	1.00
3	3	0	90.705	1.0827	1.0831	0.00067	0.045	1.00
4	1	1	95.272	1.0425	1.0406	-0.00328	-0.224	1.00
3	1	2	96.014	1.0364	1.0276	-0.01600	-1.095	1.00
4	2	0	97.173	1.0271	1.0275	0.00081	0.056	1.00
4	2	1	105.695	0.9703	0.9688	-0.00332	-0.234	1.00
1	0	3	106.015	0.9644	0.9481	-0.03739	-2.663	1.00
1	1	3	109.402	0.9438	0.9285	-0.03730	-2.712	1.00
4	0	2	116.222	0.9072	0.9013	-0.01607	-1.225	1.00
5	1	0	117.522	0.9009	0.9012	0.00085	0.065	1.00
2	1	2	120.054	0.8892	1.1866	0.55453	39.100	1.00
4	3	1	122.733	0.8774	0.8763	-0.00326	-0.264	1.00
3	3	2	123.655	0.8738	0.8685	-0.01612	-1.327	1.00
4	2	2	131.841	0.8437	0.8390	-0.01572	-1.451	1.00
3	0	3	136.542	0.8292	0.8188	-0.03703	-3.796	1.00
5	2	1	140.044	0.8196	0.8188	-0.00301	-0.320	1.00
4	4	0	143.108	0.8120	0.8123	0.00128	0.144	1.00
5	3	0	155.856	0.7877	0.7881	0.00158	0.262	1.00

Table 4. Rutile non-linear least-squares output

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 10.45634 B= 12.84264 C= 24.46974
+/- 0.0031 0.0049 0.0074

SSR (DEGREES 2-THETA): 5.3031D-02
RMS RESIDUALS (DEGREES 2-THETA): 3.3954D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	1	11.498	7.6900	7.6970	0.00003	0.010	1.00
1	1	3	15.370	5.7600	5.7505	-0.00010	-0.025	1.00
0	2	2	15.588	5.6900	5.6858	0.00006	0.016	1.00
2	0	2	19.469	4.8000	4.8076	0.00014	0.029	1.00
1	1	5	21.187	4.1900	4.1899	-0.00000	-0.000	1.00
2	2	0	21.873	4.0600	4.0543	-0.00017	-0.031	1.00
1	3	1	22.723	3.9100	3.9108	0.00003	0.005	1.00
2	2	2	23.082	3.8500	3.8485	-0.00005	-0.009	1.00
1	3	3	24.921	3.5700	3.5636	-0.00028	-0.045	1.00
0	2	6	25.879	3.4400	3.4426	0.00013	0.020	1.00
2	2	4	26.346	3.3800	3.3795	-0.00003	-0.004	1.00
3	1	1	26.749	3.3300	3.3324	0.00013	0.020	1.00
2	0	6	27.769	3.2100	3.2156	0.00034	0.050	1.00
2	1	5	28.680	3.1100	3.1097	-0.00002	-0.003	1.00
1	3	5	28.966	3.0800	3.0792	-0.00005	-0.007	1.00
0	0	8	29.159	3.0600	3.0587	-0.00009	-0.013	1.00
0	4	4	31.452	2.8420	2.8429	0.00008	0.010	1.00
3	3	1	33.305	2.6880	2.6865	-0.00015	-0.019	1.00
2	4	2	33.497	2.6730	2.6700	-0.00031	-0.039	1.00
1	3	7	34.182	2.6210	2.6212	0.00002	0.002	1.00
4	0	0	34.276	2.6140	2.6141	0.00001	0.001	1.00
3	3	3	34.896	2.5690	2.5657	-0.00039	-0.047	1.00
2	4	4	35.976	2.5010	2.4975	-0.00044	-0.051	1.00
3	1	7	37.057	2.4240	2.4238	-0.00002	-0.003	1.00
4	0	4	37.376	2.4040	2.4038	-0.00003	-0.003	1.00
4	2	2	37.850	2.3750	2.3751	0.00001	0.001	1.00
3	3	5	37.999	2.3660	2.3660	0.00000	0.000	1.00
0	2	10	39.347	2.2880	2.2866	-0.00024	-0.026	1.00
0	4	8	40.700	2.2150	2.2146	-0.00007	-0.008	1.00
1	1	11	42.070	2.1460	2.1453	-0.00015	-0.015	1.00
3	1	9	42.780	2.1120	2.1145	0.00053	0.053	1.00
2	2	10	43.080	2.0980	2.0950	-0.00066	-0.065	1.00
5	1	1	43.083	2.0570	2.0568	-0.00005	-0.005	1.00
0	0	12	44.346	2.0410	2.0391	-0.00044	-0.043	1.00
3	5	3	45.233	2.0030	2.0043	0.00033	0.031	1.00
4	2	8	45.594	1.9880	1.9872	-0.00020	-0.019	1.00
2	4	8	46.358	1.9570	1.9554	-0.00043	-0.040	1.00
4	4	4	47.149	1.9260	1.9242	-0.00049	-0.046	1.00
3	5	5	47.834	1.9000	1.9047	0.00136	0.125	1.00
3	1	11	49.041	1.8560	1.8555	-0.00016	-0.015	1.00
1	5	9	49.554	1.8380	1.8380	0.00001	0.001	1.00
2	4	10	49.989	1.8230	1.8239	0.00030	0.027	1.00
2	5	8	51.253	1.7810	1.7818	0.00028	0.025	1.00
5	3	5	52.100	1.7540	1.7542	0.00007	0.006	1.00
6	0	2	53.044	1.7250	1.7253	0.00012	0.010	1.00
1	3	13	53.955	1.6980	1.7001	0.00088	0.074	1.00

Table 5. Sulfur non-linear least-squares output

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	1	1	7.6936	5	1	1	2.0556	4	6	2	1.6405								
0	0	4	6.1150	2	4	8	2.0385	2	4	12	1.6344								
1	1	3	5.7481	0	0	12	2.0383	6	2	4	1.6208								
0	2	2	5.6844	1	5	7	2.0299	3	7	1	1.6194								
2	0	2	4.8049	4	4	0	2.0262	5	5	1	1.6174								
1	1	5	4.1882	3	5	3	2.0036	0	6	10	1.6106								
2	2	0	4.0525	5	1	3	1.9998	4	0	12	1.6071								
1	3	1	3.9098	4	4	2	1.9990	0	8	0	1.6050								
2	2	2	3.8468	4	0	8	1.9862	1	7	7	1.6048								
1	3	3	3.5626	2	6	0	1.9803	2	2	14	1.6044								
0	2	6	3.4415	2	6	2	1.9549	6	0	6	1.6016								
2	2	4	3.3780	1	3	11	1.9390												
3	1	1	3.3305	4	4	4	1.9234												
2	0	6	3.2141	3	3	9	1.9160												
0	4	0	3.2100	3	5	5	1.9040												
1	1	7	3.2088	5	1	5	1.9008												
3	1	3	3.1080	4	2	8	1.8975												
1	3	5	3.0783	0	6	6	1.8948												
0	0	8	3.0575	2	6	4	1.8840												
2	2	6	2.8741	5	3	1	1.8725												
0	4	4	2.8422	3	1	11	1.8546												
3	1	5	2.7707	1	5	9	1.8375												
3	3	1	2.6853	1	1	13	1.8328												
2	4	2	2.6691	5	3	3	1.8301												
1	3	7	2.6203	2	4	10	1.8233												
4	0	0	2.6125	2	2	12	1.8210												
1	3	9	2.5768	4	4	6	1.8145												
3	3	3	2.5645	1	7	1	1.8018												
2	4	4	2.4967	2	6	6	1.7813												
1	5	1	2.4809	3	5	7	1.7790												
2	3	8	2.4407	5	1	7	1.7764												
3	1	7	2.4226	1	7	3	1.7639												
4	0	4	2.4024	5	3	5	1.7533												
1	5	3	2.3847	6	0	2	1.7243												
4	2	2	2.3738	0	4	12	1.7207												
3	3	5	2.3650	4	2	10	1.7203												
0	2	10	2.2857	3	3	11	1.7169												
2	4	6	2.2713	1	3	13	1.6995												
4	2	4	2.2501	1	7	5	1.6948												
1	3	9	2.2409	4	4	8	1.6890												
1	5	5	2.2218	0	2	14	1.6858												
2	0	10	2.2153	6	2	0	1.6809												
0	4	8	2.2139	6	2	2	1.6653												
1	1	11	2.1444	2	6	8	1.6621												
3	3	7	2.1373	1	5	11	1.6597												
3	1	9	2.1135	2	0	14	1.6570												
0	6	2	2.1080	5	3	7	1.6543												
2	2	10	2.0941	3	5	9	1.6452												
4	2	6	2.0809	5	1	9	1.6431												
3	5	1	2.0597	3	1	13	1.6419												

Table 6. hkl generator (Sulfur)

. REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 10.4553A +/- 0.0025 B= 12.8480A 0.0043 C= 24.4683A 0.0063

SSR (DEGREES 2-THETA): 3.8367D-02
RMS RESIDUALS (DEGREES 2-THETA): 2.8880D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	1/D**2	RESIDUALS 2-THETA	WEIGHT
1	1	1	11.498	7.6900	7.6977	0.00003	0.012	1.00
1	1	3	15.370	5.7600	5.7507	-0.00010	-0.025	1.00
0	2	2	15.588	5.6800	5.6876	0.00008	0.021	1.00
2	0	2	18.469	4.8000	4.8072	0.00013	0.028	1.00
1	1	5	21.187	4.1900	4.1899	-0.00000	-0.001	1.00
2	2	0	21.873	4.0600	4.0547	-0.00016	-0.029	1.00
1	3	1	22.723	3.9100	3.9121	0.00007	0.012	1.00
2	2	2	23.082	3.8500	3.8489	-0.00004	-0.007	1.00
1	3	3	24.921	3.5700	3.5646	-0.00024	-0.039	1.00
0	2	6	25.879	3.4400	3.4429	0.00014	0.022	1.00
2	2	4	26.346	3.3800	3.3797	-0.00002	-0.003	1.00
3	1	1	26.749	3.3300	3.3322	0.00012	0.018	1.00
2	0	6	27.769	3.2100	3.2154	0.00033	0.048	1.00
3	1	3	28.680	3.1100	3.1095	-0.00003	-0.005	1.00
1	3	5	28.966	3.0800	3.0798	-0.00001	-0.002	1.00
0	0	8	29.159	3.0600	3.0585	-0.00010	-0.014	1.00
0	4	4	31.452	2.8420	2.8438	0.00016	0.020	1.00
3	3	1	33.305	2.6880	2.6868	-0.00012	-0.015	1.00
2	4	2	33.497	2.6730	2.6707	-0.00024	-0.030	1.00
1	3	7	34.182	2.6210	2.6215	0.00005	0.007	1.00
4	0	0	34.276	2.6140	2.6138	-0.00002	-0.002	1.00
3	3	3	34.896	2.5690	2.5659	-0.00037	-0.043	1.00
2	4	4	35.876	2.5010	2.4981	-0.00037	-0.043	1.00
3	1	7	37.057	2.4240	2.4237	-0.00004	-0.005	1.00
4	0	4	37.376	2.4040	2.4036	-0.00006	-0.006	1.00
4	2	2	37.850	2.3750	2.3750	0.00001	0.001	1.00
3	3	5	37.999	2.3660	2.3662	0.00003	0.003	1.00
0	2	10	39.347	2.2880	2.2866	-0.00024	-0.025	1.00
0	4	8	40.700	2.2150	2.2150	-0.00000	-0.000	1.00
1	1	11	42.070	2.1460	2.1452	-0.00017	-0.017	1.00
3	1	9	42.780	2.1120	2.1144	0.00050	0.051	1.00
2	2	10	43.080	2.0980	2.0949	-0.00066	-0.066	1.00
5	1	1	43.983	2.0570	2.0566	-0.00009	-0.009	1.00
0	0	12	44.346	2.0410	2.0390	-0.00047	-0.045	1.00
3	5	3	45.233	2.0030	2.0048	0.00044	0.042	1.00
4	0	8	45.594	1.9880	1.9871	-0.00024	-0.023	1.00
2	6	2	46.358	1.9570	1.9561	-0.00025	-0.024	1.00
4	4	4	47.149	1.9260	1.9244	-0.00044	-0.041	1.00
5	1	5	47.834	1.9000	1.9017	0.00049	0.045	1.00
3	1	11	49.041	1.8560	1.8554	-0.00020	-0.018	1.00
1	5	9	49.554	1.8380	1.8384	0.00012	0.011	1.00
2	4	10	49.989	1.8230	1.8241	0.00035	0.031	1.00
2	6	6	51.253	1.7810	1.7823	0.00045	0.039	1.00
5	3	5	52.100	1.7540	1.7542	0.00007	0.006	1.00
6	0	2	53.044	1.7250	1.7251	0.00006	0.005	1.00
1	3	13	53.955	1.6980	1.7002	0.00089	0.075	1.00

Table 7. Sulfur non-linear least-squares output (re-indexed)

SYLVITE 4-587

CUBIC

LAMBDA = 1.540562A; INPUT DATA: 13 VALUES OF D(OBS)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 1/D**2

A = 6.2930A
+/- 0.0002

SSR (DEGREES 2-THETA): 3.6665D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.6794D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA	WEIGHT
2	0	0	28.336	3.1470	3.1465	-0.00003	-0.004	1.00
2	2	0	40.528	2.2240	2.2249	0.00017	0.018	1.00
2	2	2	50.195	1.8160	1.8166	0.00022	0.019	1.00
4	0	0	58.640	1.5730	1.5733	0.00013	0.011	1.00
4	2	0	66.386	1.4070	1.4072	0.00012	0.009	1.00
4	2	2	73.726	1.2840	1.2846	0.00053	0.038	1.00
4	4	0	87.629	1.1126	1.1125	-0.00020	-0.013	1.00
6	0	0	94.496	1.0490	1.0488	-0.00028	-0.019	1.00
6	2	0	101.442	0.9951	0.9950	-0.00017	-0.012	1.00
6	2	2	108.587	0.9486	0.9487	0.00026	0.019	1.00
4	4	4	116.000	0.9083	0.9083	0.00006	0.005	1.00
6	4	0	123.925	0.8727	0.8727	-0.00004	-0.003	1.00
6	4	2	132.671	0.8410	0.8409	-0.00019	-0.018	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A = 6.2931A
+/- 0.0002

SSR (DEGREES 2-THETA): 3.6648D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.6790D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA	WEIGHT
2	0	0	28.336	3.1470	3.1465	-0.00003	-0.004	1.00
2	2	0	40.528	2.2240	2.2249	0.00017	0.018	1.00
2	2	2	50.195	1.8160	1.8166	0.00022	0.019	1.00
4	0	0	58.640	1.5730	1.5733	0.00014	0.011	1.00
4	2	0	66.386	1.4070	1.4072	0.00012	0.009	1.00
4	2	2	73.726	1.2840	1.2846	0.00053	0.038	1.00
4	4	0	87.629	1.1126	1.1125	-0.00019	-0.013	1.00
6	0	0	94.496	1.0490	1.0488	-0.00027	-0.019	1.00
6	2	0	101.442	0.9951	0.9950	-0.00016	-0.011	1.00
6	2	2	108.587	0.9486	0.9487	0.00027	0.019	1.00
4	4	4	116.000	0.9083	0.9083	0.00007	0.005	1.00
6	4	0	123.925	0.8727	0.8727	-0.00003	-0.002	1.00
6	4	2	132.671	0.8410	0.8409	-0.00018	-0.017	1.00

Table 8. sylvite non-linear least-squares output

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= [illegible]A C= 29.5534A
+/- [illegible] 0.0102

SSR (DEGREES 2-THETA): 6.9678D-01
RMS RESIDUALS (DEGREES 2-THETA): 1.2176D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
0	0	6	19.230	4.8600	4.9256	0.00112	0.245	1.00
1	0	1	24.640	3.6100	3.6393	0.00123	0.201	1.00
0	0	8	27.334	3.2600	3.2837	0.00135	0.201	1.00
0	1	5	28.775	3.1000	3.1162	0.00108	0.152	1.00
1	0	7	32.460	2.7560	2.7686	0.00120	0.152	1.00
0	1	8	34.576	2.5920	2.6026	0.00121	0.145	1.00
0	0	10	36.603	2.4530	2.4628	0.00132	0.151	1.00
1	0	9	39.276	2.2920	2.3011	0.00150	0.162	1.00
0	1	11	41.805	2.1590	2.1673	0.00164	0.167	1.00
1	1	0	42.902	2.1110	2.1173	0.00132	0.133	1.00
0	0	12	45.158	1.9650	1.9702	0.00137	0.130	1.00
1	0	11	47.071	1.9290	1.9322	0.00089	0.092	1.00
0	1	12	49.931	1.8250	1.8295	0.00148	0.131	1.00
1	1	9	51.407	1.7760	1.7794	0.00122	0.106	1.00
2	0	5	52.454	1.7430	1.7513	0.00310	0.266	1.00
0	2	7	54.616	1.6790	1.6818	0.00119	0.100	1.00
0	1	13	56.102	1.6380	1.6419	0.00175	0.143	1.00
1	1	12	57.439	1.6030	1.6055	0.00122	0.098	1.00
0	1	14	59.804	1.5690	1.5709	0.00097	0.077	1.00
0	2	10	59.845	1.5560	1.5581	0.00110	0.087	1.00
2	0	11	61.209	1.5130	1.5145	0.00087	0.067	1.00
1	1	15	64.626	1.4410	1.4423	0.00090	0.067	1.00
0	2	13	65.390	1.4260	1.4272	0.00084	0.063	1.00
0	0	22	66.386	1.4070	1.4073	0.00022	0.016	1.00
1	2	2	67.692	1.3830	1.3800	-0.00227	-0.167	1.00
0	1	20	68.480	1.3690	1.3706	0.00124	0.090	1.00
1	2	5	69.609	1.3480	1.3495	0.00119	0.086	1.00
1	1	18	72.968	1.2970	1.2975	0.00042	0.030	1.00
2	1	10	75.513	1.2580	1.2549	-0.00312	-0.219	1.00
0	0	24	77.398	1.2320	1.2314	-0.00065	-0.045	1.00
3	0	0	79.151	1.2220	1.2224	0.00044	0.030	1.00
0	1	23	78.921	1.2120	1.2126	0.00073	0.050	1.00
2	1	13	81.170	1.1840	1.1834	-0.00067	-0.046	1.00
1	1	21	82.179	1.1720	1.1720	0.00003	0.002	1.00
2	1	14	88.086	1.1080	1.1086	0.00094	0.064	1.00
0	2	22	89.306	1.0960	1.0946	-0.00217	-0.148	1.00
0	3	3	90.459	1.0850	1.0836	-0.00213	-0.145	1.00
2	2	3	94.024	1.0530	1.0523	-0.00124	-0.085	1.00
1	0	29	98.734	1.0150	1.0143	-0.00133	-0.092	1.00
3	1	5	100.345	1.0030	1.0024	-0.00126	-0.087	1.00
0	3	6	102.730	0.9861	0.9851	-0.00206	-0.144	1.00
2	2	12	104.712	0.9728	0.9726	-0.00048	-0.033	1.00
1	3	1	106.348	0.9623	0.9617	-0.00127	-0.090	1.00
1	2	23	109.626	0.9425	0.9423	-0.00045	-0.032	1.00
0	2	28	114.569	0.9150	0.9148	-0.00065	-0.049	1.00
3	1	9	115.446	0.9061	0.9060	-0.00036	-0.028	1.00
4	0	7	118.563	0.8960	0.8959	-0.00023	-0.018	1.00

Table 9. Tetradymite non-linear least-squares output

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
0	0	3	9.8630	0	2	11	1.5160	0	2	20	1.1518	2	2	12	0.9735				
0	0	6	4.9315	1	1	15	1.4438	3	0	9	1.1466	1	1	27	0.9734				
1	0	1	3.6424	1	0	19	1.4336	0	3	9	1.1466	2	0	26	0.9672				
1	0	1	3.6424	0	1	19	1.4336	1	0	25	1.1264	0	2	26	0.9672				
1	0	2	3.5623	2	0	13	1.4286	0	1	25	1.1264	2	1	22	0.9656				
1	0	2	3.5623	0	2	13	1.4286	2	1	16	1.1097	1	2	22	0.9656				
1	0	4	3.2878	0	0	21	1.4090	1	2	16	1.1097	3	1	10	0.9626				
1	0	4	3.2878	2	1	1	1.3857	3	0	12	1.0959	1	3	10	0.9626				
1	0	4	3.2877	1	2	1	1.3857	0	3	12	1.0959	3	1	11	0.9521				
1	0	5	3.1191	2	0	14	1.3857	0	0	27	1.0959	1	3	11	0.9521				
1	0	5	3.1191	0	2	14	1.3857	1	0	26	1.0870	2	1	23	0.9433				
1	0	7	2.7714	2	1	2	1.3812	0	1	26	1.0870	1	2	23	0.9433				
1	0	7	2.7714	1	2	2	1.3812	2	1	17	1.0848	2	2	15	0.9334				
1	0	8	2.6053	1	0	20	1.3722	1	2	17	1.0848	3	1	13	0.9293				
1	0	8	2.6053	0	1	20	1.3722	2	0	22	1.0848	1	3	13	0.9293				
1	0	12	2.4657	2	1	4	1.3635	0	2	22	1.0848	3	0	21	0.9238				
1	0	10	2.3036	1	2	4	1.3635	1	1	24	1.0656	0	3	21	0.9238				
1	0	10	2.3036	2	1	5	1.3506	2	2	0	1.0595	1	0	31	0.9238				
1	0	11	2.1696	1	2	5	1.3506	2	2	3	1.0535	0	1	31	0.9238				
1	0	11	2.1696	2	1	7	1.3181	2	0	23	1.0534	4	0	1	0.9171				
1	0	13	2.1190	1	2	7	1.3181	0	2	23	1.0534	0	4	1	0.9171				
1	0	13	2.0718	2	0	16	1.3026	3	0	15	1.0397	3	1	14	0.9171				
1	0	15	1.9726	0	2	16	1.3026	0	3	15	1.0397	1	3	14	0.9171				
1	0	16	1.9469	2	1	8	1.2989	2	2	6	1.0359	4	0	2	0.9158				
1	0	13	1.9343	1	2	8	1.2989	2	1	19	1.0359	0	4	2	0.9158				
1	0	13	1.9343	1	1	18	1.2988	1	2	19	1.0359	2	0	28	0.9158				
1	0	1	1.8316	2	0	17	1.2629	3	1	1	1.0174	0	2	28	0.9158				
1	0	1	1.8316	0	2	17	1.2629	1	3	1	1.0174	4	0	4	0.9106				
1	0	14	1.8315	1	0	22	1.2628	3	1	2	1.0156	0	4	4	0.9106				
1	0	14	1.8315	0	1	22	1.2628	1	3	2	1.0156	4	0	5	0.9067				
1	0	2	1.8212	2	1	10	1.2560	1	0	28	1.0155	0	4	5	0.9067				
1	0	2	1.8212	1	2	10	1.2560	0	1	28	1.0155	2	1	25	0.9004				
1	0	4	1.7812	2	1	11	1.2329	2	1	20	1.0120	1	2	25	0.9004				
1	0	4	1.7812	1	2	11	1.2329	1	2	20	1.0120	4	0	7	0.8967				
1	0	9	1.7811	0	0	24	1.2329	3	1	4	1.0085	0	4	7	0.8967				
1	0	5	1.7528	2	0	0	1.2234	1	3	4	1.0085	1	0	32	0.8966				
1	0	5	1.7528	2	0	3	1.2141	2	2	9	1.0084	0	1	32	0.8966				
1	0	7	1.6934	0	2	3	1.2141	3	1	5	1.0032	0	0	33	0.8966				
1	0	7	1.6934	1	0	23	1.2141	1	3	5	1.0032	1	1	30	0.8942				
1	0	16	1.6515	0	1	23	1.2141	2	0	25	0.9946	3	1	16	0.8918				
1	0	16	1.6515	3	0	6	1.1874	0	2	25	0.9946	1	3	16	0.8918				
1	0	8	1.6439	0	3	6	1.1874	3	1	7	0.9897	2	0	29	0.8918				
1	0	8	1.6439	2	0	19	1.1874	1	3	7	0.9897	0	2	29	0.8918				
1	0	18	1.6438	0	2	19	1.1874	0	0	30	0.9863	4	0	8	0.8906				
1	0	12	1.6071	2	1	13	1.1846	1	0	29	0.9830	0	4	8	0.8906				
1	0	17	1.5727	1	2	13	1.1846	0	1	29	0.9830	2	2	18	0.8906				
1	0	17	1.5727	1	1	21	1.1733	3	1	8	0.9815								
1	0	10	1.5596	2	1	14	1.1597	1	3	8	0.9815								
2	0	10	1.5596	1	2	14	1.1597	3	0	18	0.9814								
2	0	11	1.5160	2	0	20	1.1518	0	3	18	0.9814								

Table 10. hkl generator (Tetradymite)

SOLUTION MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 8.3942A +/- 0.0008 B= 8.7925A 0.0006 C= 4.6486A 0.0003

SSR (DEGREES 2-THETA): 8.1713D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.2417D-02

	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
	20.188	4.3950	4.3962	0.00003	0.006	1.00
	21.166	4.1940	4.1971	0.00008	0.016	1.00
	21.599	4.1110	4.1096	-0.00004	-0.008	1.00
	22.866	3.8963	3.8945	-0.00005	-0.009	1.00
	24.078	3.6930	3.6910	-0.00008	-0.013	1.00
	27.962	3.1950	3.1941	-0.00006	-0.008	1.00
	29.385	3.0370	3.0358	-0.00009	-0.012	1.00
	29.899	2.9860	2.9853	-0.00005	-0.007	1.00
	30.409	2.9370	2.9364	-0.00005	-0.007	1.00
	36.184	2.4804	2.4792	-0.00016	-0.018	1.00
	37.496	2.3966	2.3973	0.00010	0.011	1.00
	37.795	2.3783	2.3777	-0.00009	-0.010	1.00
	38.085	2.3609	2.3605	-0.00006	-0.007	1.00
	38.701	2.3247	2.3243	-0.00006	-0.007	1.00
	38.905	2.3130	2.3129	-0.00002	-0.002	1.00
	40.096	2.2470	2.2471	0.00002	0.002	1.00
	41.012	2.1989	2.1981	-0.00015	-0.015	1.00
	41.561	2.1711	2.1707	-0.00008	-0.009	1.00
	42.466	2.1269	2.1264	-0.00010	-0.010	1.00
	42.932	2.1049	2.1047	-0.00004	-0.004	1.00
	44.017	2.0555	2.0548	-0.00016	-0.016	1.00
	45.613	1.9872	1.9872	-0.00001	-0.001	1.00
	45.749	1.9816	1.9810	-0.00014	-0.014	1.00
	46.610	1.9470	1.9472	0.00006	0.006	1.00
	46.942	1.9340	1.9337	-0.00008	-0.007	1.00
	48.675	1.8691	1.8690	-0.00004	-0.004	1.00
	49.061	1.8553	1.8556	0.00010	0.009	1.00
	50.042	1.8212	1.8211	-0.00003	-0.002	1.00
	50.767	1.7969	1.7960	-0.00030	-0.027	1.00
	51.296	1.7796	1.7797	0.00004	0.004	1.00
	54.914	1.6706	1.6706	0.00002	0.001	1.00
	55.436	1.6551	1.6562	0.00004	0.003	1.00
	56.770	1.6203	1.6201	-0.00007	-0.006	1.00
	57.502	1.6014	1.6018	0.00018	0.014	1.00
	57.660	1.5974	1.5970	-0.00017	-0.014	1.00
	58.392	1.5791	1.5790	-0.00004	-0.003	1.00
	58.829	1.5684	1.5689	0.00026	0.021	1.00
	59.290	1.5572	1.5576	0.00016	0.013	1.00
	59.412	1.5544	1.5542	-0.00013	-0.010	1.00
	59.636	1.5491	1.5495	0.00023	0.018	1.00
	60.305	1.5335	1.5337	0.00013	0.010	1.00
	60.615	1.5264	1.5260	-0.00022	-0.017	1.00
	60.986	1.5180	1.5179	-0.00007	-0.005	1.00
	61.770	1.5006	1.5014	0.00048	0.037	1.00
	62.138	1.4926	1.4926	0.00002	0.002	1.00
	62.444	1.4860	1.4861	0.00004	0.003	1.00
	63.457	1.4647	1.4654	0.00045	0.034	1.00
	63.617	1.4614	1.4614	0.00001	0.000	1.00
	64.511	1.4433	1.4436	0.00018	0.014	1.00
	64.687	1.4399	1.4398	-0.00003	-0.002	1.00
	64.960	1.4344	1.4342	-0.00016	-0.012	1.00
	65.806	1.4180	1.4179	-0.00005	-0.004	1.00
	66.638	1.4023	1.4024	0.00004	0.003	1.00

Table 11. Topaz non-linear least-squares output

URANINITE 5-550

CUBIC

LAMBDA = 1.540562A; INPUT DATA: 16 VALUES OF D(OBS)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 1/D**2

A= 5.4684A
+/- 0.0002

SSR (DEGREES 2-THETA): 6.3216D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.9877D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA	WEIGHT
1	1	1	28.244	3.1570	3.1572	0.00001	0.002	1.00
2	0	0	32.716	2.7350	2.7342	-0.00008	-0.010	1.00
2	2	0	46.942	1.9340	1.9334	-0.00018	-0.016	1.00
3	1	1	55.695	1.6490	1.6488	-0.00010	-0.008	1.00
2	2	2	58.396	1.5790	1.5786	-0.00021	-0.017	1.00
4	0	0	68.537	1.3680	1.3671	-0.00071	-0.052	1.00
3	3	1	75.725	1.2550	1.2545	-0.00047	-0.033	1.00
4	2	0	78.075	1.2230	1.2228	-0.00025	-0.018	1.00
4	2	2	87.265	1.1163	1.1162	-0.00010	-0.007	1.00
5	1	1	94.108	1.0523	1.0524	0.00016	0.011	1.00
4	4	0	105.670	0.9666	0.9667	0.00018	0.013	1.00
5	3	1	112.892	0.9243	0.9243	0.00007	0.005	1.00
6	0	0	115.379	0.9114	0.9114	-0.00001	-0.000	1.00
6	2	0	125.976	0.8646	0.8646	0.00009	0.007	1.00
5	3	3	134.949	0.8339	0.8339	0.00007	0.007	1.00
6	2	2	138.284	0.8243	0.8244	0.00032	0.033	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 5.4683A
+/- 0.0002

SSR (DEGREES 2-THETA): 6.2390D-03
RMS RESIDUALS (DEGREES 2-THETA): 1.9747D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA	WEIGHT
1	1	1	28.244	3.1570	3.1571	0.00001	0.001	1.00
2	0	0	32.716	2.7350	2.7342	-0.00008	-0.010	1.00
2	2	0	46.942	1.9340	1.9333	-0.00018	-0.017	1.00
3	1	1	55.695	1.6490	1.6488	-0.00011	-0.009	1.00
2	2	2	58.396	1.5790	1.5786	-0.00022	-0.018	1.00
4	0	0	68.537	1.3680	1.3671	-0.00072	-0.053	1.00
3	3	1	75.725	1.2550	1.2545	-0.00049	-0.034	1.00
4	2	0	78.075	1.2230	1.2228	-0.00027	-0.019	1.00
4	2	2	87.265	1.1163	1.1162	-0.00013	-0.009	1.00
5	1	1	94.108	1.0523	1.0524	0.00013	0.009	1.00
4	4	0	105.670	0.9666	0.9667	0.00015	0.011	1.00
5	3	1	112.892	0.9243	0.9243	0.00003	0.002	1.00
6	0	0	115.379	0.9114	0.9114	-0.00004	-0.003	1.00
6	2	0	125.976	0.8646	0.8646	0.00005	0.004	1.00
5	3	3	134.949	0.8339	0.8339	0.00003	0.003	1.00
6	2	2	138.284	0.8243	0.8244	0.00028	0.028	1.00

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 10.3319A C= 7.3230A
+/- 0.0057 0.0046

SSR (DEGREES 2-THETA): 2.4698D-01
RMS RESIDUALS (DEGREES 2-THETA): 8.1701D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	0	9.875	8.9500	8.9477	-0.00001	-0.003	1.00
1	0	1	15.588	5.6800	5.6670	-0.00014	-0.036	1.00
1	1	0	17.170	5.1600	5.1659	0.00009	0.020	1.00
2	0	0	19.846	4.4700	4.4738	0.00009	0.017	1.00
1	1	1	21.034	4.2200	4.2213	0.00003	0.006	1.00
2	0	1	23.266	3.8200	3.8177	-0.00008	-0.014	1.00
0	0	2	24.164	3.6800	3.6615	-0.00075	-0.124	1.00
1	0	2	26.346	3.3800	3.3887	0.00045	0.069	1.00
2	1	1	29.062	3.0700	3.0703	0.00002	0.003	1.00
3	0	0	29.878	2.9880	2.9826	-0.00041	-0.056	1.00
2	0	2	31.520	2.8360	2.8335	-0.00022	-0.028	1.00
3	0	1	32.411	2.7600	2.7622	0.00021	0.027	1.00
2	2	0	34.756	2.5790	2.5830	0.00046	0.055	1.00
2	1	2	36.115	2.4850	2.4843	-0.00009	-0.010	1.00
2	2	1	36.883	2.4350	2.4359	0.00012	0.014	1.00
1	0	3	38.251	2.3510	2.3549	0.00060	0.066	1.00
3	0	2	38.905	2.3130	2.3125	-0.00009	-0.010	1.00
4	0	0	40.283	2.2370	2.2369	-0.00001	-0.002	1.00
1	1	3	40.796	2.2100	2.2070	-0.00055	-0.058	1.00
2	2	2	42.802	2.1110	2.1106	-0.00008	-0.008	1.00
3	1	2	44.050	2.0540	2.0543	0.00006	0.006	1.00
2	1	3	45.788	1.9800	1.9793	-0.00019	-0.018	1.00
4	1	0	46.534	1.9500	1.9525	0.00068	0.064	1.00
4	0	2	47.568	1.9100	1.9089	-0.00032	-0.030	1.00
3	0	3	48.103	1.8900	1.8890	-0.00030	-0.027	1.00
0	0	4	49.583	1.8370	1.8307	-0.00203	-0.181	1.00
1	0	4	51.007	1.7890	1.7936	0.00160	0.140	1.00
3	1	3	52.649	1.7370	1.7402	0.00123	0.105	1.00
4	1	2	53.110	1.7230	1.7229	-0.00005	-0.004	1.00
2	0	4	54.162	1.6920	1.6944	0.00098	0.082	1.00
3	3	1	54.757	1.6750	1.6763	0.00054	0.045	1.00
4	0	3	55.769	1.6470	1.6492	0.00097	0.080	1.00
2	1	4	57.167	1.6100	1.6100	-0.00001	-0.001	1.00
2	2	3	58.928	1.5660	1.5711	0.00262	0.208	1.00
4	2	2	60.067	1.5390	1.5351	-0.00212	-0.166	1.00
4	1	3	60.457	1.5300	1.5248	-0.00294	-0.230	1.00
2	2	4	62.073	1.4940	1.4936	-0.00023	-0.017	1.00

Table 13. Vanadinite non-linear least-squares output

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
1	0	0	8.9469	2	2	3	1.7766												
1	0	1	5.6761	3	1	3	1.7426												
1	1	0	5.1655	1	3	3	1.7426												
2	0	0	4.4735	5	0	1	1.7385												
1	1	1	4.2249	1	1	4	1.7298												
2	0	1	3.8203	4	1	2	1.7238												
0	0	2	3.6715	1	4	2	1.7238												
1	0	2	3.3966	3	3	0	1.7218												
2	1	0	3.3816	2	0	4	1.6983												
1	2	0	3.3816	4	2	0	1.6908												
2	1	1	3.0716	2	4	0	1.6908												
1	2	1	3.0716	3	3	1	1.6764												
2	1	2	2.9926	4	0	3	1.6512												
3	0	0	2.9823	4	2	1	1.6477												
2	0	2	2.8380	2	4	1	1.6477												
3	0	1	2.7631	2	1	4	1.6134												
2	2	0	2.5827	1	2	4	1.6134												
2	1	2	2.4873	5	0	2	1.6085												
1	2	2	2.4873	5	1	0	1.6069												
3	1	0	2.4814	1	5	0	1.6069												
1	3	0	2.4814	3	2	3	1.5728												
2	2	1	2.4364	2	3	3	1.5728												
1	0	3	2.3609	5	1	1	1.5698												
3	1	1	2.3508	1	5	1	1.5698												
1	3	1	2.3508	3	0	4	1.5633												
3	0	2	2.3149	3	3	2	1.5589												
4	0	0	2.2367	4	2	2	1.5358												
1	1	3	2.2119	2	4	2	1.5358												
2	0	3	2.1473	4	1	3	1.5263												
4	0	1	2.1397	1	4	3	1.5263												
2	2	2	2.1124	2	2	4	1.4963												
3	1	2	2.0559	6	0	0	1.4912												
1	3	2	2.0559	3	1	4	1.4758												
3	2	0	2.0526	1	3	4	1.4758												
2	3	0	2.0526	5	1	2	1.4721												
2	1	3	1.9828	1	5	2	1.4721												
1	2	3	1.9828	4	3	0	1.4709												
3	2	1	1.9768	3	4	0	1.4709												
2	3	1	1.9768	6	0	1	1.4613												
4	1	0	1.9524	1	0	5	1.4492												
1	4	0	1.9524	5	0	3	1.4445												
4	0	2	1.9102	4	3	1	1.4422												
3	0	3	1.8920	3	4	1	1.4422												
4	1	1	1.8868	5	2	0	1.4327												
1	4	1	1.8868	2	5	0	1.4327												
0	0	4	1.8357	4	0	4	1.4190												
1	0	4	1.7983	1	1	5	1.4126												
3	2	2	1.7916	3	3	3	1.4083												
2	3	2	1.7916	5	2	1	1.4061												
5	0	0	1.7894	2	5	1	1.4061												

Table 14. hkl generator (Vanadinite)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 10.3300A C= 7.3442A
+/- 0.0031 0.0039

SSR (DEGREES 2-THETA): 1.1233D-01
RMS RESIDUALS (DEGREES 2-THETA): 5.5100D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	0	9.875	8.9500	8.9461	-0.00001	-0.004	1.00
1	0	1	15.588	5.6800	5.6764	-0.00004	-0.010	1.00
1	1	0	17.170	5.1600	5.1650	0.00007	0.017	1.00
2	0	0	19.846	4.4700	4.4730	0.00007	0.014	1.00
1	1	1	21.034	4.2200	4.2248	0.00013	0.024	1.00
2	0	1	23.266	3.8200	3.8202	0.00001	0.002	1.00
0	0	2	24.164	3.6800	3.6721	-0.00032	-0.053	1.00
2	1	0	26.346	3.3800	3.3813	0.00007	0.010	1.00
2	1	1	29.062	3.0700	3.0714	0.00010	0.014	1.00
3	0	0	29.878	2.9880	2.9820	-0.00045	-0.061	1.00
2	0	2	31.520	2.8360	2.8382	0.00019	0.025	1.00
3	0	1	32.411	2.7600	2.7629	0.00028	0.036	1.00
2	2	0	34.756	2.5790	2.5825	0.00041	0.049	1.00
2	1	2	36.115	2.4850	2.4874	0.00031	0.036	1.00
2	2	1	36.383	2.4350	2.4363	0.00018	0.020	1.00
3	1	1	38.251	2.3510	2.3507	-0.00005	-0.006	1.00
3	0	2	38.905	2.3130	2.3149	0.00030	0.033	1.00
4	0	0	40.283	2.2370	2.2365	-0.00009	-0.009	1.00
1	1	3	40.796	2.2100	2.2122	0.00040	0.042	1.00
2	2	2	42.802	2.1110	2.1124	0.00030	0.030	1.00
3	1	2	44.050	2.0540	2.0559	0.00043	0.042	1.00
2	1	3	45.788	1.9900	1.9829	0.00075	0.071	1.00
4	1	0	46.534	1.9500	1.9522	0.00059	0.055	1.00
4	0	2	47.568	1.9100	1.9101	0.00004	0.003	1.00
3	0	3	48.103	1.8900	1.8921	0.00063	0.058	1.00
0	0	4	49.583	1.8370	1.8360	-0.00031	-0.028	1.00
5	0	0	51.007	1.7890	1.7892	0.00008	0.007	1.00
5	0	1	52.649	1.7370	1.7384	0.00052	0.045	1.00
4	1	2	53.110	1.7230	1.7237	0.00029	0.025	1.00
4	2	0	54.162	1.6920	1.6906	-0.00056	-0.047	1.00
3	1	3	54.757	1.6750	1.6762	0.00052	0.044	1.00
4	2	1	55.769	1.6470	1.6476	0.00025	0.021	1.00
5	0	2	57.167	1.6100	1.6084	-0.00075	-0.060	1.00
3	0	4	58.928	1.5660	1.5635	-0.00133	-0.105	1.00
4	2	2	60.067	1.5390	1.5357	-0.00182	-0.142	1.00
4	1	3	60.457	1.5300	1.5263	-0.00207	-0.162	1.00
2	2	4	62.073	1.4940	1.4964	0.00144	0.111	1.00

Table 15. Vanadinite non-linear least-squares output (re-indexed)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 9.[illegible]236A B= 17.3370A C= 6.[illegible]349A
+/- 0.0020 0.0036 0.0014

SSR (DEGREES 2-THETA): 2.0530D-02
RMS RESIDUALS (DEGREES 2-THETA): 2.1601D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
0	2	0	10.194	8.6700	8.6685	-0.00000	-0.002	1.00
1	1	0	10.498	8.4200	8.4142	-0.00002	-0.007	1.00
1	2	1	15.671	5.6500	5.6529	0.00003	0.008	1.00
1	1	1	16.463	5.3800	5.3744	-0.00007	-0.017	1.00
1	3	0	17.905	4.9500	4.9543	0.00007	0.016	1.00
2	0	0	18.430	4.8100	4.8118	0.00003	0.007	1.00
0	4	0	20.446	4.3400	4.3342	-0.00014	-0.027	1.00
1	3	1	21.983	4.0400	4.0410	0.00003	0.006	1.00
2	0	1	22.433	3.9600	3.9625	0.00008	0.015	1.00
2	1	1	23.022	3.8600	3.8629	0.00010	0.018	1.00
0	1	2	26.033	3.4200	3.4237	0.00018	0.029	1.00
2	4	0	27.681	3.2200	3.2204	0.00002	0.004	1.00
2	1	0	28.217	3.1600	3.1543	-0.00036	-0.052	1.00
1	2	2	29.062	3.0700	3.0701	0.00001	0.001	1.00
1	5	1	30.209	2.9560	2.9557	-0.00003	-0.003	1.00
2	4	1	30.537	2.9250	2.9245	-0.00004	-0.005	1.00
0	6	0	30.916	2.8900	2.8895	-0.00004	-0.006	1.00
3	3	0	31.878	2.8050	2.8047	-0.00002	-0.003	1.00
2	5	1	34.371	2.6070	2.6094	0.00027	0.033	1.00
1	6	1	34.840	2.5730	2.5729	-0.00002	-0.002	1.00
2	3	2	35.321	2.5390	2.5390	0.00000	0.000	1.00
2	6	0	36.266	2.4750	2.4772	0.00029	0.033	1.00
4	0	0	37.344	2.4060	2.4059	-0.00002	-0.002	1.00
3	0	2	38.049	2.3630	2.3625	-0.00007	-0.009	1.00
4	0	1	39.581	2.2750	2.2747	-0.00005	-0.005	1.00
3	5	1	40.358	2.2330	2.2313	-0.00030	-0.032	1.00
0	6	2	40.452	2.2280	2.2263	-0.00031	-0.032	1.00
4	2	1	41.029	2.1980	2.2002	0.00042	0.044	1.00
1	2	3	41.166	2.1910	2.1896	-0.00026	-0.027	1.00
1	6	2	41.643	2.1670	2.1690	0.00040	0.041	1.00
1	3	3	42.930	2.1050	2.1072	0.00047	0.047	1.00
2	0	3	43.145	2.0950	2.0958	0.00018	0.018	1.00
2	1	3	43.450	2.0810	2.0807	-0.00007	-0.007	1.00
3	6	1	44.096	2.0520	2.0522	0.00004	0.004	1.00
2	2	3	44.392	2.0390	2.0371	-0.00044	-0.043	1.00
2	6	2	44.785	2.0220	2.0205	-0.00036	-0.035	1.00
2	8	0	45.886	1.9760	1.9760	-0.00001	-0.001	1.00
3	7	0	46.283	1.9600	1.9604	0.00011	0.010	1.00
1	9	0	48.130	1.8890	1.8889	-0.00004	-0.004	1.00
3	6	2	49.814	1.8290	1.8290	-0.00000	-0.000	1.00
5	2	1	50.255	1.8140	1.8145	0.00015	0.013	1.00
3	3	3	50.915	1.7920	1.7915	-0.00019	-0.016	1.00
5	3	1	51.688	1.7670	1.7667	-0.00010	-0.009	1.00
0	0	4	52.357	1.7460	1.7462	0.00008	0.007	1.00

Table 16. Wavellite non-linear least-squares output

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A =	5.[illegible]44A	B=	8.7928A	C=	6.[illegible]795A
+/-	0.0588		0.1006		0.0780

SSR (DEGREES 2-THETA): 1.0382D+02
RMS RESIDUALS (DEGREES 2-THETA): 1.6529D+00

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	0	19.450	4.5600	4.5293	-0.00065	-0.133	1.00
0	2	0	19.936	4.4500	4.3964	-0.00124	-0.245	1.00
1	1	1	23.901	3.7200	3.6932	-0.00105	-0.176	1.00
2	0	1	24.231	3.6700	3.6200	-0.00206	-0.340	1.00
0	2	2	27.725	3.2150	3.1998	-0.00154	-0.224	1.00
0	1	2	29.504	3.0250	2.9985	-0.00194	-0.266	1.00
1	0	2	32.545	2.7490	2.7308	-0.00177	-0.223	1.00
2	2	0	33.718	2.6560	2.6422	-0.00149	-0.182	1.00
1	1	2	34.089	2.6280	2.6079	-0.00224	-0.270	1.00
1	3	0	34.604	2.5900	2.5631	-0.00315	-0.375	1.00
2	2	0	39.473	2.2810	2.2647	-0.00278	-0.297	1.00
0	4	0	40.499	2.2260	2.1982	-0.00513	-0.535	1.00
2	2	1	41.988	2.1500	2.1342	-0.00322	-0.326	1.00
0	4	1	42.951	2.1040	2.0783	-0.00562	-0.558	1.00
2	0	2	44.186	2.0480	2.0348	-0.00311	-0.302	1.00
1	3	2	44.855	2.0190	1.9980	-0.00519	-0.498	1.00
1	1	3	46.788	1.9400	1.9249	-0.00418	-0.389	1.00
2	2	2	48.057	1.8590	1.8466	-0.00390	-0.351	1.00
0	4	2	49.785	1.8300	1.8100	-0.00663	-0.588	1.00
3	1	1	52.640	1.7370	1.7271	-0.00379	-0.324	1.00
2	4	0	53.682	1.7060	1.6898	-0.00660	-0.555	1.00
3	1	1	54.687	1.6770	1.6671	-0.00422	-0.351	1.00
1	3	3	55.695	1.6490	1.6366	-0.00560	-0.460	1.00
1	5	1	56.289	1.6330	1.6143	-0.00874	-0.712	1.00
2	3	3	59.052	1.5630	1.5502	-0.00678	-0.536	1.00
0	4	3	59.895	1.5430	1.5284	-0.00807	-0.632	1.00
3	3	0	60.853	1.5210	1.5098	-0.00645	-0.501	1.00
2	4	2	61.434	1.5080	1.4932	-0.00873	-0.674	1.00
0	6	0	62.538	1.4840	1.4655	-0.01155	-0.882	1.00
3	3	2	68.140	1.3750	1.3646	-0.00806	-0.590	1.00
1	3	4	68.651	1.3660	1.3541	-0.00944	-0.688	1.00
3	1	3	69.699	1.3480	1.3407	-0.00605	-0.438	1.00
2	4	2	70.478	1.3350	1.4932	0.11262	8.370	1.00
4	0	0	70.905	1.3280	1.3211	-0.00594	-0.427	1.00
2	6	0	72.998	1.2950	1.2815	-0.01258	-0.892	1.00
3	4	1	76.226	1.2480	1.3230	0.07072	5.011	1.00
0	2	5	77.323	1.2330	1.2253	-0.00825	-0.574	1.00
2	5	3	78.688	1.2150	1.2058	-0.01034	-0.716	1.00

Table 17. Witherite non-linear least-squares output

H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D	H	K	L	D
0	1	1	5.2129	0	2	4	1.5120	1	2	5	1.2034								
0	2	0	4.4520	2	4	2	1.5072	2	6	2	1.2017								
1	0	1	4.0962	1	5	2	1.4949												
1	1	1	3.7213	0	6	0	1.4840												
0	2	2	3.2150	1	4	3	1.4826												
1	2	1	3.0144	3	3	1	1.4802												
1	0	2	2.7567	2	5	0	1.4793												
0	3	1	2.6948	3	2	2	1.4650												
2	0	0	2.6570	2	3	3	1.4542												
1	1	2	2.6282	1	2	4	1.4542												
0	2	2	2.6064	2	5	1	1.4416												
2	1	0	2.5461	1	6	1	1.3953												
2	0	1	2.4556	2	0	4	1.3754												
1	3	1	2.4034	3	3	2	1.3749												
2	1	1	2.3672	0	5	3	1.3697												
1	2	2	2.3401	1	3	4	1.3660												
2	2	0	2.2816	3	0	3	1.3654												
0	4	0	2.2260	2	1	4	1.3593												
2	2	1	2.1502	3	4	1	1.3549												
2	1	3	2.0838	3	1	3	1.3496												
2	0	2	2.0481	0	6	2	1.3474												
1	3	2	2.0175	2	5	2	1.3439												
2	1	2	1.9960	2	4	3	1.3349												
1	0	3	1.9877	4	0	0	1.3285												
2	3	0	1.9796	1	5	3	1.3264												
1	4	1	1.9559	2	2	4	1.3141												
1	1	3	1.9400	4	1	0	1.3140												
2	3	1	1.8920	1	6	2	1.3061												
2	2	2	1.8606	3	2	3	1.3054												
0	4	2	1.8301	0	4	4	1.3032												
1	2	3	1.8150	4	0	1	1.3010												
0	3	3	1.7376	2	6	0	1.2956												
1	4	2	1.7304	4	1	1	1.2874												
0	5	1	1.7162	4	2	0	1.2730												
3	0	1	1.7077	3	4	2	1.2728												
2	4	0	1.7063	0	1	5	1.2728												
2	3	2	1.6857	2	6	1	1.2701												
3	1	1	1.6772	1	4	4	1.2657												
2	0	3	1.6682	1	0	5	1.2499												
1	3	3	1.6516	4	2	1	1.2488												
2	4	1	1.6492	2	3	4	1.2479												
2	1	3	1.6397	0	7	1	1.2478												
1	5	1	1.6331	3	3	3	1.2404												
0	3	4	1.6075	1	1	5	1.2378												
3	2	1	1.5944	3	5	1	1.2326												
2	2	3	1.5621	4	0	2	1.2278												
3	0	2	1.5514	2	5	3	1.2175												
1	0	4	1.5386	4	1	2	1.2163												
3	1	2	1.5284	1	7	1	1.2148												
1	1	4	1.5162	4	3	0	1.2126												

Table 18. hkl generator (Witherite)

LEAST-SQUARES REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF 2-THETA

A= 5.[illegible]125A B= 8.9034A C= 6.[illegible]367A
+/- [illegible].24 0.0031 0.[illegible]28

SSR (DEGREES 2-THETA): 1.3823D-01
RMS RESIDUALS (DEGREES 2-THETA): 6.0313D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	RESIDUALS 2-THETA	WEIGHT
1	[illegible]	[illegible]	19.450	4.5600	4.5621	0.00004	0.009	1.00
[illegible]	[illegible]	[illegible]	19.936	4.4500	4.4517	0.00004	0.008	1.00
1	1	[illegible]	23.901	3.7200	3.7220	0.00008	0.013	1.00
[illegible]	[illegible]	[illegible]	24.231	3.6700	3.6613	-0.00035	-0.058	1.00
[illegible]	[illegible]	[illegible]	27.725	3.2150	3.2183	0.00020	0.029	1.00
1	[illegible]	1	29.504	3.0250	3.0147	-0.00075	-0.103	1.00
1	[illegible]	[illegible]	32.545	2.7490	2.7526	0.00035	0.044	1.00
2	[illegible]	[illegible]	33.718	2.6560	2.6562	0.00003	0.003	1.00
1	[illegible]	[illegible]	34.088	2.6280	2.6298	0.00020	0.024	1.00
[illegible]	[illegible]	[illegible]	34.604	2.5900	2.6081	0.00207	0.248	1.00
[illegible]	[illegible]	[illegible]	39.473	2.2810	2.2810	0.00001	0.001	1.00
[illegible]	[illegible]	[illegible]	40.490	2.2260	2.2259	-0.00003	-0.003	1.00
[illegible]	[illegible]	1	41.988	2.1500	2.1500	0.00001	0.001	1.00
[illegible]	[illegible]	[illegible]	42.951	2.1040	2.1036	-0.00008	-0.008	1.00
[illegible]	[illegible]	[illegible]	44.186	2.0480	2.0486	0.00014	0.014	1.00
1	[illegible]	[illegible]	44.855	2.0190	2.0182	-0.00020	-0.019	1.00
1	[illegible]	[illegible]	46.788	1.9400	1.9415	0.00042	0.040	1.00
[illegible]	[illegible]	[illegible]	48.957	1.8590	1.8610	0.00062	0.056	1.00
[illegible]	[illegible]	[illegible]	49.785	1.8300	1.8307	0.00022	0.019	1.00
[illegible]	[illegible]	[illegible]	52.649	1.7370	1.7388	0.00067	0.057	1.00
[illegible]	[illegible]	[illegible]	53.682	1.7060	1.7061	0.00002	0.002	1.00
[illegible]	1	1	54.687	1.6770	1.6768	-0.00007	-0.006	1.00
[illegible]	1	1	55.695	1.6490	1.6491	0.00005	0.004	1.00
1	1	1	56.289	1.6330	1.6331	0.00005	0.004	1.00
1	[illegible]	[illegible]	59.052	1.5630	1.5628	-0.00008	-0.007	1.00
1	[illegible]	[illegible]	59.895	1.5430	1.5401	-0.00160	-0.126	1.00
1	1	[illegible]	60.853	1.5210	1.5175	-0.00198	-0.154	1.00
[illegible]	[illegible]	[illegible]	61.434	1.5080	1.5074	-0.00038	-0.029	1.00
[illegible]	[illegible]	[illegible]	62.538	1.4840	1.4839	-0.00006	-0.004	1.00
[illegible]	[illegible]	[illegible]	65.140	1.3750	1.3749	-0.00005	-0.004	1.00
[illegible]	[illegible]	[illegible]	68.651	1.3660	1.3670	0.00076	0.056	1.00
[illegible]	[illegible]	[illegible]	69.699	1.3480	1.3476	-0.00036	-0.026	1.00
[illegible]	[illegible]	[illegible]	70.478	1.3350	1.3354	0.00029	0.021	1.00
[illegible]	[illegible]	[illegible]	70.905	1.3280	1.3281	0.00010	0.007	1.00
[illegible]	[illegible]	[illegible]	72.996	1.2950	1.2955	0.00042	0.030	1.00
[illegible]	[illegible]	4	76.226	1.2480	1.2486	0.00060	0.042	1.00
[illegible]	[illegible]	1	77.323	1.2330	1.2324	-0.00064	-0.044	1.00
1	[illegible]	1	78.688	1.2150	1.2147	-0.00030	-0.021	1.00

Table Witherite non-linear least-squares output (reindexed)

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A=	5.3221A	B=	8.8986A	C=	6.4380A
+/-	0.0053		0.0068		0.0062

SSR (DEGREES 2-THETA): 6.5084D-01
RMS RESIDUALS (DEGREES 2-THETA): 1.3087D-01

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	1	0	19.450	4.5600	4.5675	0.00016	0.032	1.00
0	2	0	19.950	4.4469	4.4493	0.00005	0.011	1.00
1	1	1	23.850	3.7278	3.7252	-0.00010	-0.017	1.00
0	2	1	24.250	3.6672	3.6602	-0.00028	-0.047	1.00
0	0	2	27.750	3.2121	3.2190	0.00041	0.060	1.00
1	2	1	29.550	3.0204	3.0158	-0.00033	-0.046	1.00
1	0	2	32.550	2.7486	2.7544	0.00056	0.070	1.00
2	0	0	33.720	2.6558	2.6611	0.00056	0.068	1.00
1	1	2	34.050	2.6308	2.6312	0.00004	0.005	1.00
0	2	2	34.620	2.5888	2.6080	0.00219	0.263	1.00
2	2	0	39.500	2.2795	2.2838	0.00072	0.077	1.00
0	4	0	40.550	2.2229	2.2246	0.00032	0.034	1.00
2	2	1	42.050	2.1470	2.1524	0.00108	0.110	1.00
0	4	1	43.000	2.1017	2.1026	0.00020	0.020	1.00
2	0	2	44.200	2.0474	2.0510	0.00083	0.081	1.00
1	3	2	44.850	2.0192	2.0184	-0.00021	-0.020	1.00
1	1	3	46.750	1.9415	1.9423	0.00022	0.020	1.00
2	2	2	48.950	1.8593	1.8626	0.00104	0.094	1.00
0	4	2	49.800	1.8295	1.8301	0.00020	0.018	1.00
0	3	3	52.700	1.7354	1.7387	0.00123	0.105	1.00
2	4	0	53.750	1.7040	1.7068	0.00112	0.095	1.00
3	1	1	54.700	1.6766	1.6795	0.00124	0.103	1.00
2	4	1	55.750	1.6475	1.6498	0.00102	0.084	1.00
1	5	1	56.350	1.6314	1.6327	0.00059	0.049	1.00
2	2	3	59.100	1.5619	1.5639	0.00106	0.084	1.00
1	0	4	59.900	1.5429	1.5406	-0.00126	-0.099	1.00
1	1	4	60.900	1.5199	1.5180	-0.00110	-0.086	1.00
2	4	2	61.500	1.5065	1.5079	0.00081	0.063	1.00
0	6	0	62.600	1.4827	1.4831	0.00026	0.020	1.00
3	3	2	68.180	1.3743	1.3763	0.00156	0.115	1.00
1	3	4	68.680	1.3655	1.3672	0.00132	0.096	1.00
0	6	2	69.650	1.3488	1.3470	-0.00149	-0.108	1.00
2	4	3	69.820	1.3460	1.3358	-0.00843	-0.609	1.00
4	0	0	70.550	1.3338	1.3305	-0.00278	-0.201	1.00
2	6	0	73.000	1.2950	1.2955	0.00046	0.033	1.00
2	3	4	76.200	1.2484	1.2491	0.00078	0.054	1.00
3	5	1	77.220	1.2344	1.2332	-0.00130	-0.091	1.00
1	7	1	78.750	1.2142	1.2143	0.00006	0.004	1.00

Table 20, Witherite non-linear least-squares output
(38 values of 2-theta from diffraction pattern)

REFINEME MINIMIZING THE SUM OF THE SQUARES OF TH RESIDUALS OF THETA

A= 5.4347A C= 12.1105A
+/- 0.0003 0.0013

SSR (DEGREES 2-THETA): 2.4658D-02
RMS RESIDUALS (DEGREES 2-THETA): 2.5145D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	1	17.868	4.9600	4.9583	-0.00003	-0.006	1.00
1	1	2	27.472	3.2440	3.2446	0.00004	0.005	1.00
0	0	4	29.474	3.0280	3.0276	-0.00003	-0.004	1.00
2	0	0	32.926	2.7180	2.7173	-0.00007	-0.008	1.00
2	1	1	37.718	2.3830	2.3829	-0.00001	-0.001	1.00
1	0	5	40.758	2.2120	2.2123	0.00006	0.006	1.00
2	1	3	43.428	2.0820	2.0822	0.00004	0.004	1.00
2	0	4	44.808	2.0210	2.0223	0.00031	0.030	1.00
2	2	0	47.305	1.9200	1.9214	0.00041	0.038	1.00
1	1	6	51.068	1.7870	1.7869	-0.00002	-0.002	1.00
3	0	3	55.549	1.6530	1.6528	-0.00010	-0.009	1.00
2	2	4	56.705	1.6220	1.6223	0.00015	0.012	1.00
0	0	8	61.119	1.5150	1.5138	-0.00068	-0.053	1.00
3	2	1	61.981	1.4960	1.4958	-0.00014	-0.011	1.00
3	2	3	66.174	1.4110	1.4121	0.00077	0.057	1.00
4	0	0	69.055	1.3590	1.3587	-0.00027	-0.019	1.00
2	0	8	71.220	1.3229	1.3224	-0.00039	-0.028	1.00
3	1	6	72.126	1.3085	1.3085	0.00002	0.002	1.00
3	2	5	73.982	1.2802	1.2797	-0.00044	-0.031	1.00
3	3	2	75.832	1.2535	1.2532	-0.00028	-0.019	1.00
4	0	4	76.807	1.2400	1.2396	-0.00045	-0.031	1.00
4	2	0	78.680	1.2151	1.2152	0.00014	0.010	1.00
2	2	8	80.766	1.1889	1.1891	0.00024	0.017	1.00
4	1	5	83.446	1.1574	1.1578	0.00047	0.032	1.00
1	1	10	83.658	1.1550	1.1551	0.00007	0.005	1.00
3	1	8	85.441	1.1354	1.1360	0.00077	0.053	1.00
4	2	4	86.166	1.1277	1.1278	0.00010	0.007	1.00
3	3	6	90.845	1.0814	1.0815	0.00023	0.016	1.00
5	1	2	94.414	1.0497	1.0497	-0.00002	-0.001	1.00
4	0	8	99.264	1.0110	1.0111	0.00027	0.019	1.00
2	1	11	100.345	1.0030	1.0029	-0.00027	-0.019	1.00
3	1	10	102.167	0.9900	0.9900	-0.00010	-0.007	1.00
5	2	3	103.701	0.9795	0.9791	-0.00094	-0.065	1.00
4	4	0	106.571	0.9609	0.9607	-0.00040	-0.029	1.00
4	2	8	108.773	0.9475	0.9477	0.00037	0.027	1.00
5	1	6	109.608	0.9426	0.9425	-0.00025	-0.018	1.00
5	3	2	113.476	0.9212	0.9212	-0.00003	-0.002	1.00
4	4	4	114.552	0.9156	0.9157	0.00033	0.024	1.00
6	0	0	116.528	0.9057	0.9058	0.00021	0.016	1.00

Table 21. Wulf ıite non-linear least-squares output

REFINEMENT MINIMIZING THE SUM OF THE SQUARES OF THE RESIDUALS OF THETA

A= 6.[illegible]37A C= 5.9791A
+/- 0.0003 0.0004

SSR (DEGREES 2-THETA): 1.2834D-02
RMS RESIDUALS (DEGREES 2-THETA): 1.8140D-02

H	K	L	2-THETA	D(OBS)	D(CALC)	RESIDUALS 1/D**2	2-THETA	WEIGHT
1	0	1	20.009	4.4340	4.4323	-0.00004	-0.008	1.00
2	0	0	26.980	3.3020	3.3019	-0.00001	-0.001	1.00
2	1	1	33.796	2.6500	2.6479	-0.00023	-0.028	1.00
1	1	2	35.626	2.5180	2.5178	-0.00003	-0.004	1.00
2	2	0	38.506	2.3360	2.3348	-0.00019	-0.021	1.00
2	0	2	40.662	2.2170	2.2161	-0.00016	-0.016	1.00
3	0	1	43.781	2.0660	2.0657	-0.00007	-0.007	1.00
1	0	3	47.621	1.9080	1.9080	0.00001	0.001	1.00
3	2	1	52.196	1.7510	1.7512	0.00008	0.007	1.00
3	1	2	53.478	1.7120	1.7120	-0.00001	-0.001	1.00
4	0	0	55.622	1.6510	1.6509	-0.00003	-0.003	1.00
4	1	1	59.725	1.5470	1.5471	0.00005	0.004	1.00
0	0	4	62.027	1.4950	1.4948	-0.00013	-0.010	1.00
4	2	0	62.868	1.4770	1.4766	-0.00023	-0.017	1.00
3	3	2	67.803	1.3810	1.3806	-0.00031	-0.023	1.00
2	0	4	68.881	1.3620	1.3617	-0.00021	-0.015	1.00
4	3	1	73.327	1.2900	1.2897	-0.00032	-0.023	1.00
2	2	4	75.442	1.2590	1.2589	-0.00012	-0.008	1.00
4	1	3	76.226	1.2480	1.2485	0.00047	0.033	1.00
5	1	2	80.815	1.1883	1.1884	0.00009	0.006	1.00
4	4	0	82.590	1.1672	1.1674	0.00023	0.016	1.00
4	0	4	88.096	1.1079	1.1081	0.00025	0.017	1.00
6	0	0	88.834	1.1006	1.1006	0.00003	0.002	1.00
6	1	1	92.291	1.0682	1.0682	-0.00004	-0.003	1.00
5	3	2	93.332	1.0590	1.0591	0.00013	0.009	1.00
4	2	4	94.308	1.0506	1.0505	-0.00018	-0.012	1.00
5	2	3	95.068	1.0442	1.0444	0.00038	0.026	1.00
3	2	5	100.551	1.0015	1.0013	-0.00040	-0.028	1.00
1	1	6	104.453	0.9745	0.9746	0.00016	0.011	1.00
6	3	1	104.941	0.9713	0.9713	0.00010	0.007	1.00
4	1	5	107.005	0.9582	0.9582	0.00002	0.001	1.00
6	1	3	107.821	0.9532	0.9534	0.00041	0.029	1.00
7	0	1	111.460	0.9321	0.9319	-0.00060	-0.044	1.00
4	4	4	113.685	0.9201	0.9200	-0.00013	-0.010	1.00
5	4	3	114.533	0.9157	0.9160	0.00067	0.050	1.00
3	1	6	117.838	0.8994	0.8994	-0.00009	-0.007	1.00
5	5	2	119.544	0.8915	0.8914	-0.00022	-0.017	1.00
6	0	4	120.708	0.8863	0.8863	-0.00004	-0.004	1.00
5	2	5	128.252	0.8561	0.8561	0.00012	0.011	1.00

Table 22. Zircon non-linear least-squares output

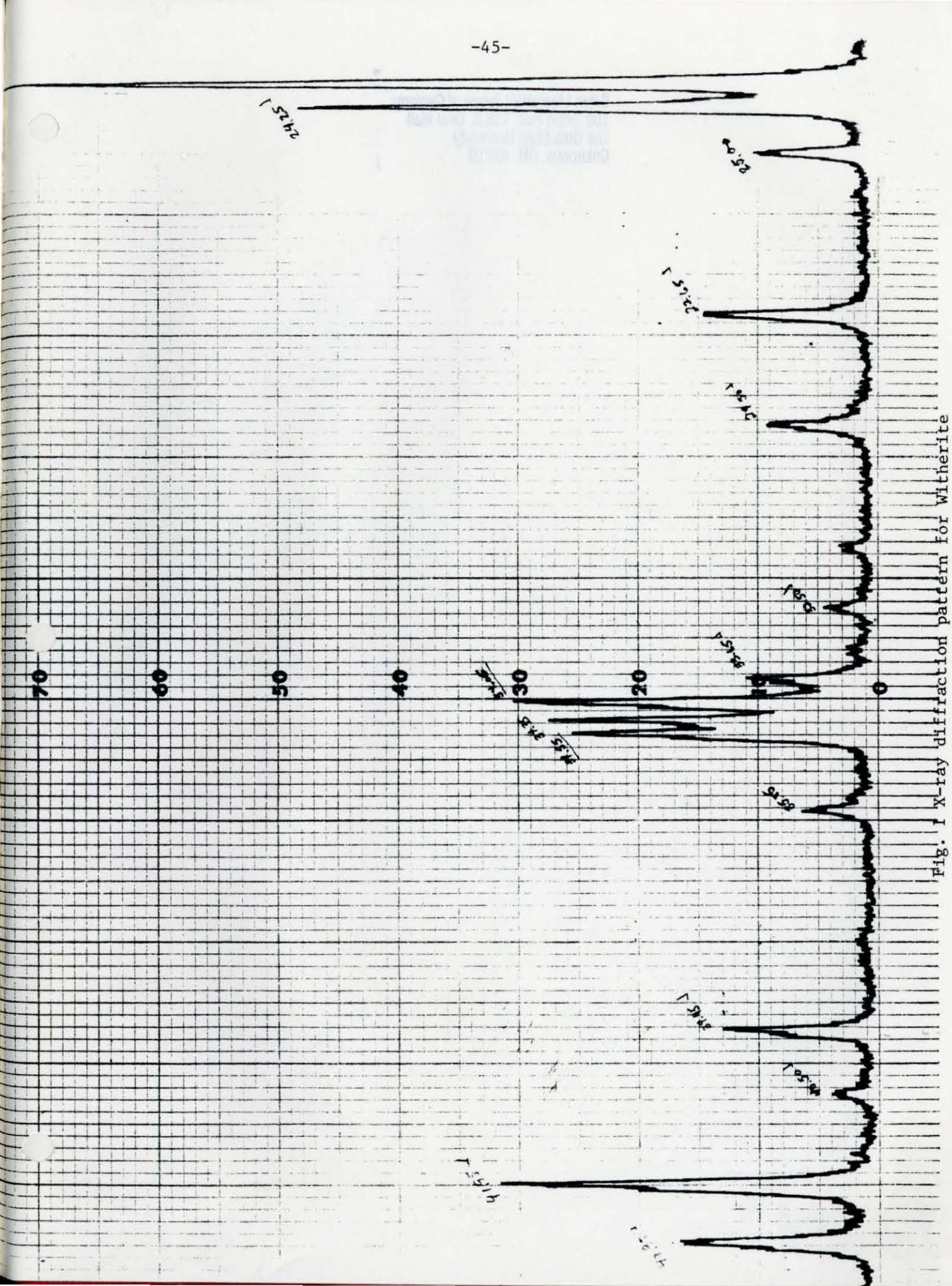

Fig. 1 X-ray diffraction pattern for Witherite